

### SAMPLING AND ANALYSIS PLAN BREMO POWER STATION 504 OUTFALL AND PROCESS

Dominion Resources Bremo Power Station Bremo Bluff, VA



Prepared For: Virginia Electric and Power Company

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June 2016

Project No. 15-20347.220

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### **Attachments**

Attachment A -	<b>VPDES</b>	Permit	VA0004138	Section I.A.9
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Attachment B – Sampling Logs Attachment C – Standard Operating Procedure – pH Analysis Attachment D – Standard Operating Procedure – TRC Analysis

Attachment E – Manufacturers' Manuals

Attachment F – Golder-Dominion Purchase Order Agreement





Golder Associates Inc. (Golder) has prepared this Sampling and Analysis Plan (SAP) to support effluent monitoring requirements in Section I.A.9 of the Virginia Pollutant Discharge Elimination System (VPDES) Permit Number VA0004138 (see Attachment A). Monitoring activities will be conducted at the Bremo Power Station (Station) located in Bremo Bluff, Virginia, for Dominion Resources (Dominion). This SAP has been written to conform to Dominion's "Field Sampling Chemistry Guidance," as well as applicable U.S. Environmental Protection Agency (EPA) standards, and Golder Associates' Standard Operating Guidelines for Environmental Sampling.

### 1.1 Purpose

This SAP was prepared as an internal reference for the Project Team, and details the sampling and analysis activities required for the 500-series outfalls covered under the VPDES permit and process sampling to assess the effectiveness of the treatment system. Any discharge from the East, West, and North Ash Ponds, individually or in combination, as well as discharges from the Metals Cleaning Waste Pond, must meet the 500-series limitations specified in Section I.A.9 of the permit. The purpose of the compliance sampling and analysis activities is to obtain representative composite samples in accordance with VPDES Permit VA0004138, and to analyze those samples for the target constituents/parameters and Whole Effluent Toxicity (WET) characteristics. Key provisions of the SAP are addressed in the following sections:

- Types of sampling
  - Process pre and post enhanced metals treatment grab samples, composite tank samples
  - Compliance 4-hour composite, 24-hour composite, grab samples (pH only)
- Field procedures
- Sample handling
- Field documentation
- Decontamination
- Management of Investigation Derived Waste (IDW)
- Deliverables

### 1.2 Health and Safety

In addition to completing Dominion's Station-specific awareness and hazard training, project personnel will complete the field work described in this SAP in accordance with Golder's site-specific *Health and Safety/Environment Plan* (HASEP, Golder 2015), developed to address the hazards associated with the work tasks.





### 1.3 Sampling Activities

Sampling will be conducted at the 504 Outfall when the outfall is actively flowing. It is anticipated that process water from Outfalls 501, 502, 503, and 505 will be pumped to an on-site treatment system and discharged from Outfall 504. If an outfall is not discharging during the monitoring period, no sample will be collected. Compliance sampling activities will include 4-hour composite samples three times a week, monthly 4-hour composite samples, monthly 24-hour composite samples, and associated water quality measurements (field parameters).

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In addition, process system performance sampling will be conducted to determine the effectiveness of the on-site treatment system. A tank composite sample will also be collected from the effluent storage tank to characterize effluent water prior to discharge.



This section provides an overview of the types of sampling and analysis required to comply with Section I.A.9 of the permit and to assess the effectiveness of the treatment system. Specific sample collection procedures are presented in Section 3.0. Two categories of samples will be collected under this SAP: compliance samples to demonstrate conformity with the permit limits, and process samples to provide timely effluent data to track system performance. Compliance samples will be submitted to a Virginia Environmental Laboratory Accreditation Program (VELAP)-accredited laboratory for analysis under expedited turnaround time (TAT) (1 day). Golder will coordinate with the contracted laboratory(ies) to ensure TATs are met (required in Attachment F). An on-site laboratory will be established and achieve VELAP accreditation as soon as practicable to serve the project's analytical needs in a timely manner. An off-site laboratory will be used in the interim, if necessary.

In addition to the sampling activities described in this SAP, sampling procedures in this SAP may need to be modified due to field constraints, and alternative sampling procedures may need to be developed to address other types of sampling requirements. Prior to implementing a modified or new procedure, the field team will verify the appropriateness of the procedures, obtain approval from the Project Manager, and document the change to the Project Manager.

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### 2.1 Process Sampling

Golder understands that grab samples shall be collected during operation of the on-site treatment system to ensure process effectiveness. Golder will coordinate with Ground/Water Treatment Technology, LLC (GWTT) staff to collect grab samples, with Ryan to collect effluent storage tank composite samples, and with the on-site (and off-site when needed) Pace laboratory to monitor analytical results.

# S1 (pre-EMT grab) • Golder • 1 / 2 Hour • Metals

### S2 series (post-EMT grab) Golder 1 / 4 Hour (beginning when EMT is engaged)

## • Golder • 1 / Tank • Metals, Turbidity, O&G, Ammonia, Chloride

Note: EMT = Enhanced Metals Treatment

### 2.1.1 S1 and S2-series Samples – Grab Sampling Procedure

Metals

Process samples will be collected directly from a CWSTS sample port to assess the performance of system operation. Process samples will *not* be collected at the permitted Outfall 504 location, in compliance with Section II.A.3 of the permit. Process sampling will be conducted on the above noted schedule. An increase or decrease in frequency may be coordinated between Dominion and Golder, as needed. However, process samples from S1 locations must be collected at least every 4 hours to comply with the Concept Engineering Report (CER) requirements.

- Locations: During CSWTS operation, the S1 sample will be collected every 2 hours. In addition, during operation of the enhanced metals treatment (EMT), the S2-series samples will be collected every 4 hours.
  - "S1" samples will be collected after the mechanical 0.5-micron filtration, prior to the EMT to determine if basic system operation is sufficient to treat water to permit/threshold limits.
  - "S2" samples will be collected after the EMT, but prior to the effluent storage tanks, to determine if the EMT is treating water to permit/threshold limits. S2 samples will be collected during the first scheduled sampling time after the EMT is engaged.
  - "S2A through S2F" samples will be collected from the midpoint between EMT vessels 1 and 2, to detect breakthrough in the EMT media. S2A-F samples will be collected during the first scheduled sampling time after the EMT is engaged.



The following procedure will be used to sample process samples S1 (pre-EMT), S2A-F (mid-EMT), and S2 (post-EMT):

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### If Enhanced Metals Treatment (EMT) is engaged:

Samples should be collected from the cleanest location to the most impacted; therefore, process samples should be collected from S2, then S2A-F, then S1.

- 1. Designated Golder sampler will bring containment bucket, sample containers, and nitrile gloves to the S2 sample port.
- 2. Label the laboratory-supplied sample container with the correct current discharge tank, sample port ID, date, and time.
  - Tank # Year (##) Month (##) Day (##) Time (24-hour ####) S2
  - Example: Currently discharging to effluent storage tank #4, on 1/7/16 at 2:40PM collecting a sample after the EMT, label the bottle: T4-160107-1440-S2
- 3. Don new nitrile gloves.
- 4. Open sample port labelled "S2" and purge approximately 2 gallons of water to discharge into the containment bucket, to ensure the sample port is clean and representative process water is being delivered.
- 5. Fill the labeled, laboratory-provided sample container from the S2 port.
- 6. Dispose of sample gloves and bring sampling equipment to sample port S2A through S2F. Follow the procedure in steps 1-5 for each of the active EMT trains.

### If Enhanced Metals Treatment (EMT) is not engaged:

- 1. Label the laboratory-supplied sample container with the correct current discharge tank, sample port ID, date, and time.
  - Tank # Year (##) Month (##) Day (##) Time (24-hour ####) S1
  - Example: Currently discharging to effluent storage tank #4, on 1/7/16 at 2:45PM collecting a sample before the EMT, label the bottle: T4-160107-1445-S1
- 2. Don new nitrile gloves.
- 3. Open sample tap labelled "S1" and purge approximately 2 gallons of water to discharge into the containment bucket.
- 4. Fill the labeled, laboratory-provided sample container from the S1 port.
- 5. Dispose of sample gloves and place containerized purge water into influent CSWTS tank.
- 6. The sampler will immediately deliver grab sample(s) to the on-site Pace laboratory in the Bremo administration building in person to maintain chain-of-custody.
- The sampler will ensure that the Chain-of-Custody Log is signed with the date and time relinquished.





### 2.1.2 S3 Tank Sample - Composite Sampling Procedure

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Each effluent storage tank will be sampled to characterize each approximately 750,000-gallon tank of treated process water before discharge. The effluent storage tanks will be vertically composited using a dedicated, decontaminated CorePro® tank sampler (depicted below) along the approximately 11-foot water column using the following procedure. The tanks may be sampled between 80%-100% capacity.



- 1. Label the laboratory-supplied sample container(s) with the correct current discharge tank, sample ID, date, and time.
  - Tank # Year (##) Month (##) Day (##) Time (24-hour ####) S3
  - Example: Effluent storage tank #4 is 80% full, on 1/7/16 at 5:00PM, label the bottle: T4-160107-1700-S3
- 2. Place new disposable liner in carboy.
- 3. Decontaminate the CorePro® sampler using Alconox® spray followed by a deionized water rinse.
- 4. Ascend ladder to effluent storage tank with CorePro® sampler.Once near the top of the tank, don new nitrile gloves.
- 5. Submerge the sampler through the full water column allowing the bottom ball valve to collect water as it is lowered.
- 6. Once the sampler touches the base of the tank, withdraw the sampler, allowing the bottom ball valve to close.
- 7. Lower the base of the sampler into the carboy and allow the ball valve to open repeat until carboy is filled.
- Close the cap of the carboy and invert to ensure compositing is complete.
- 9. Fill the labeled, laboratory-provided sample container from the carboy.
- 10. Take pH, turbidity, ammonia, chloride, and total petroleum hydrocarbon (TPH) measurements from the sample in the carboy (see Section 2.1.3).
- 11. Dispose of sample gloves and place additional composite water into effluent storage tank.
- 12. The sampler will deliver grab sample(s) to the on-site Pace laboratory in the Bremo administration building in person to maintain chain-of-custody.
- 13. The sampler will ensure that the Chain-of-Custody Log is signed with the date and time relinquished.



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### 2.1.3 S3 Tank Sample - Field Measurement Procedures

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In addition to laboratory samples, Golder will perform field tests and record results on a Tank Composite Log. Field screening methods will have the ability to measure at or below permit limits, to detect possible non-compliant conditions within a tank prior to discharge.

### 2.1.3.1 pH Measurements

Golder will follow the Standard Operating Procedure (SOP) for pH analysis included in Attachment C.

### 2.1.3.2 **Turbidity Measurements**

Golder will follow HACH manufacturer's instructions included in Attachment E. Turbidity measurements and calibration time will be recorded on a Tank Composite Log and used to calculate total suspended solids (TSS) using a TSS-Turbidity correlation chart.

### 2.1.3.3 Chloride Measurements

Golder will follow HACH Chloride Test Kit 8-P manufacturer's instructions included in Attachment E. At least once per week, Golder will conduct a standard check with a 100 mg/L chloride standard solution, to test reagent viability.

### 2.1.3.4 Ammonia-Nitrogen Measurements

Golder will follow the La Motte Ammonia-Nitrogen 1200 Colorimeter manufacturer's manual included in Attachment E. The colorimeter will be zeroed prior to each test.

### 2.1.3.5 **TPH Measurements**

Golder will follow the HACH TPH in Water Immunoassay Method 10050 included in Attachment E. The analysis will result in absorbency values for the sample and the calibration standards (2 ppm, 5 ppm, and 10 ppm). If the sample absorbency is greater than the standard, a result of "ND" or not detected will be recorded on the Tank Composite Log. If sample absorbency values are less than the calibration standard absorbency values, the presence of TPH is indicated in the sample.

### 2.1.4 Process Sampling Laboratory Analysis

Process grab samples (S1 and S2) and tank composite sample (S3) will be analyzed by an on-site laboratory, using the DEQ-specified methods and quantification levels (QLs). In addition, S3 samples may be analyzed for a full compliance list included in Section 2.2.2.2 and Section 2.2.4.2. Samples will be analyzed for the following parameters, using similar methods as the compliance samples:

Analyte	Method	Quantification Level <sup>1</sup>
Mercury	EPA 200.8	0.1 ug/L
Total Antimony	EPA 200.8	5.0 ug/L
Total Arsenic	EPA 200.8	5.0 ug/L





Analyte	Method	Quantification Level <sup>1</sup>
Total Cadmium	EPA 200.8	1.0 ug/L
Total Chromium <sup>2</sup>	EPA 200.8	
Total Copper	EPA 200.8	5.0 ug/L
Total Lead	EPA 200.8	5.0 ug/L
Total Nickel	EPA 200.8	5.0 ug/L
Total Selenium	EPA 200.8	5.0 ug/L
Total Silver	EPA 200.8	0.4 ug/L
Total Thallium	EPA 200.8	0.2 ug/L
Total Zinc	EPA 200.8	25 ug/L

<sup>&</sup>lt;sup>1</sup>QL defined in Part I.C.1 of VPDES Permit #VA0004138

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<sup>&</sup>lt;sup>2</sup>Total chromium will be analyzed for comparison to trivalent and hexavalent chromium.



It is anticipated that process water from Outfalls 501, 502, 503, and 505 will be pumped directly to the CSWTS for treatment and be discharged from internal Outfall 504. Outfall 504 will include a shared high-density polyethylene (HDPE) pipe that is connected to a valved manifold exiting each 950,000-gallon effluent storage tank. In order to achieve discharge through Outfall 504, valves from one tank will be opened. A data logging flow meter will collect flow measurements at Outfall 504. If a discharge is present at Outfall 504, personnel from Golder will conduct the sampling activities. If no discharge is present, no samples will be collected for that monitoring period.

### Weekly 4-Hr Composite

- Golder
- 3 / Week
- Metals, TSS, O&G, Chloride, Ammonia, pH

### Monthly 4-Hr Composite

- Golder
- 1 / Month
- Metals, Free Cyanide

### Monthly 24-Hr Composite

- Golder
- 1 / Month
- WET

### 2.2.1 Compliance Auto-sampler

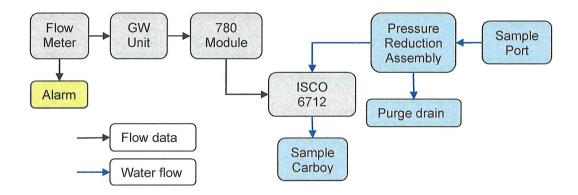


Flow-paced, composite samples will be collected at Outfall 504 to comply with permit requirements, and will consist of triweekly 4-hour composites, monthly 4-hour composites, and monthly 24-hour composites. A dedicated ISCO 6712 sampling system will be set up in a protective housing within the secondary containment structure in the effluent storage tank area. Samples will be collected directly into a lined, 4-gallon carboy, to reduce the possibility of field contamination. The carboy will be kept refrigerated using a sample refrigerator.



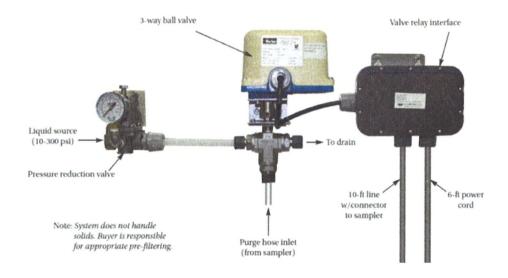
### 2.2.1.1 Auto-sampler Configuration

The system components include a pressurized line sampler assembly, data-logging flow meter, and a control module. The sampling system will be set up as indicated below.



### 2.2.1.2 Pressurized Line Sampler Assembly

Pressure in the effluent discharge pipe is expected to be greater than 15 psi. The auto-sampler has reduced functionality in pressures exceeding 15 psi. To accommodate sampling in the effluent discharge pipe, a pressurized line sampler assembly (depicted below) will be used. The assembly will be isolated with a valve to allow for troubleshooting during normal operations. A sample port in the effluent pipe will accept treated process water through the pressure reduction valve. A three-way ball valve will allow water to purge to a drain prior to each sampling aliquot, then divert fresh sample to the sample carboy.





### 2.2.1.3 Auto-sampler Programming

The dedicated auto-sampler will be programmed to collect composite samples using the following steps.

- 1. Select PROGRAM and Site Description Change NO
- 2. Number of Bottles: 1
- 3. Bottle Volume: 10,000 ml
- 4. Suction Line Length: 4 FT
- 5. FLOW PACED
- 6. Flow between sample events: 0.026 Mgal (4-Hr) and 0.078 Mgal (24-Hr)
- 7. BOTTLES/SAMPLES
- 8. Run Continuously?: NO
- 9. Bottles per Sample Event: 1
- 10. CLOCK TIME
- 11. Start Flow Count at: 60:00
- 12. Select Days: MO, WE, and FR
- 13. Programming Complete Run this Program Now?: NO

### 2.2.2 Weekly 4-Hour Composite Sampling

Three 4-hour composite samples shall be collected on a weekly basis for the combined Outfall 504. Weekly 4-hour composite samples will be collected a minimum of 48 hours apart. It is anticipated that 4-hour compliance samples will be collected on Monday, Wednesday, and Friday. During weeks with system downtime or site closures, Golder may alter this schedule to ensure sampling requirements are met.

### 2.2.2.1 Weekly 4-Hour Composite Sampling Procedure

Weekly compliance samples will be collected by Golder using a programmed ISCO 6712 auto-sampler. The auto-sampler will be programmed to collect a flow-paced 4-hour composite sample into a single lined carboy using the program in Section 2.2.1.3. The sampler will collect a 350-mL aliquot through the pressurized line each time the flow meter registers 26,000 gallons of flow. For example, at a flow of 1,300 gallons per minute, one aliquot will be collected every 20 minutes.

A 4-Hour Composite Sampling Log will be used to record composite information, water quality measurements, and sample observations. Field observations will include visual monitoring of the presence of floating solids, visible foam, or sheen as required by Section I.A.9 of the VPDES Permit.



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Golder will follow the procedure below to collect tri-weekly 4-hour composite samples.

- 1. Coordinate the courier for the date/time the sample will be completed.
- 2. Ensure that a new carboy liner is in place and the sample tube discharges into the carboy.
- 3. Start the ISCO 6712 sampler 4-hour composite program.
- Record start date and time, and sample information on 4-Hour Composite Sampling Log.
- 5. Collect a pH reading from the grab sample port and record on log following procedures in the attached pH SOP.
- 6. After 4 hours the program will stop.
- 7. Don new nitrile gloves.
- 8. Remove the carboy from the sampler and cap immediately to prevent airborne particle contamination.
- 9. Invert the carboy 3 times to ensure water is composited.
- 10. Label, date, and time the sample containers.
- 11. Fill the sample containers with the composite sample.
- 12. Record sample observations including color, presence of floating solids, and presence of visible foam.
- 13. Complete documentation on 4-Hour Composite Sampling Log and Chain-of-Custody form.
- 14. Place sample containers in a cooler and immediately transport to the on-site lab or courier.

In addition to the weekly 4-hour composite laboratory samples, a grab sample will be collected for pH, temperature, turbidity, and conductivity analysis using a field calibrated meter. The pH analysis will be performed by Golder personnel who have completed the requisite Initial Demonstration of Capability (IDC) evaluation using Golder's Standard Operating Procedure (SOP) for pH analysis (Attachment C), which conforms to EPA Method 4500 H +B.

Once per week prior to the compositing of samples, Golder will collect an Equipment Blank to indicate possible contamination sources in sampling equipment. Laboratory-provided deionized water will be poured across a new, disposable carboy liner and into pre-labelled, pre-preserved sampling containers. A new liner will then be used in the compositing process. It may be necessary to alter the equipment tested by the Equipment Blank, depending on sample collection protocol (*i.e.*, if a measuring cup is used to flow-proportion samples, the blank may be generated using the cup *in lieu* of the carboy liner).

### 2.2.2.2 Weekly 4-Hour Composite Laboratory Analysis

The weekly 4-hour composite samples will be analyzed by a VELAP-accredited laboratory (Pace), using the DEQ-specified methods and QLs. Samples will be analyzed for the following parameters:



Analyte	Method	Quantification Level <sup>1</sup>
Total Suspended Solids	SM 2540D	1.0 mg/L
Oil & Grease	EPA 1664B	5.0 mg/L
Hardness	EPA 200.8	
Chloride	SM 4500-CI-E	10 mg/L
Ammonia	EPA 350.1	0.20 mg/L
Mercury	EPA 245.1	0.1 ug/L
Total Antimony	EPA 200.8	5.0 ug/L
Total Arsenic	EPA 200.8	5.0 ug/L
Total Cadmium	EPA 200.8	1.0 ug/L
Total Chromium <sup>2</sup>	EPA 200.8	
Hexavalent Chromium <sup>2</sup>	SM 3500-Cr B	5.0 ug/L
Trivalent Chromium <sup>2</sup>	Calculation	5.0 ug/L
Total Copper	EPA 200.8	5.0 ug/L
Total Lead	EPA 200.8	5.0 ug/L
Total Nickel	EPA 200.8	5.0 ug/L
Total Selenium	EPA 200.8	5.0 ug/L
Total Silver	EPA 200.8	0.4 ug/L
Total Thallium	EPA 200.8	1.0 ug/L
Total Zinc	EPA 200.8	25 ug/L

<sup>&</sup>lt;sup>1</sup>QL defined in Part I.C.1 of VPDES Permit #VA0004138

### 2.2.3 Monthly 4-Hour Composite

Golder understands that one 4-hour composite sample shall be collected on a monthly basis for 500-series outfalls that are discharging. Monthly 4-hour composite samples will be collected during the monthly 24-hour WET sampling event as required in Section I.A.9.i of the VPDES permit. During weeks with holidays or other closures, Golder may alter the schedule to ensure sampling requirements are met, as a full operational week is needed to sample for WET analysis.

### 2.2.3.1 Monthly 4-Hour Sampling Procedure

The manual procedure will be used for the monthly 4-hour composite as presented in Section 2.2.5 in order to simultaneously collect a 4-hour and 24-hour composite.

Once per month prior to the compositing of samples, Golder will collect an Equipment Blank to indicate possible contamination sources in sampling equipment. Laboratory-provided deionized water will be poured across a new disposable carboy liner and into pre-labelled, pre-preserved sampling containers. A

<sup>&</sup>lt;sup>2</sup>Total chromium and hexavalent chromium are used to calculate trivalent chromium. Only hexavalent chromium and trivalent chromium will be reported in the analytical package, as required by the permit.



new liner will then be used in the compositing process. The Equipment Blank will be analyzed for monthly metals and free cyanide as required by the permit.

A 4-Hour Composite Sampling Log will be used to record composite information, water quality measurements, and sample observations. Field observations will include visual monitoring of the presence of floating solids or visible foam as required by Section I.A.9 of the VPDES Permit.

### 2.2.3.2 Monthly 4-Hour Sampling Laboratory Analysis

The monthly 4-hour composite samples will be analyzed by a VELAP-accredited laboratory, using the DEQ-specified methods and QLs. Samples will be analyzed for the following parameters:

Analyte	Method	Quantification Level <sup>1</sup>
Free Cyanide	OIA-1677 or ASTM 4282-02 (once VELAP certified)	
Aluminum	200.7	
Barium	200.7	
Beryllium	200.7	
Boron	200.7	
Cobalt	200.7	
Iron	200.7	0.25 mg/L
Molybdenum	200.7	
Vanadium	200.7	

<sup>&</sup>lt;sup>1</sup>QL defined in Part I.C.1 of VPDES Permit #VA0004138

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### 2.2.4 Monthly WET Sampling

Golder understands that monthly 24-hour composite samples for Whole Effluent Toxicity (WET) analysis are required by the VPDES Permit for internal Outfall 504. If a discharge is present at Outfall 504, personnel from Golder will conduct the sampling activities. It is preferred that the samples be collected in the first week of each month, to allow for resampling should unproductive test results warrant resampling to maintain compliance. Golder will coordinate with Coastal Bioanalysts, Inc. (CBI), the selected laboratory, to determine an appropriate schedule.

### 2.2.4.1 Monthly WET Sampling Procedure

The monthly WET composite samples (set of 3) will be collected using a programmed ISCO 6712 Autosampler, or equivalent using the procedure in Section 2.2.2.1.

The composited sample will be placed into pre-labeled, laboratory-supplied sample containers. The samples will be placed on ice in a cooler for preservation, and shipped/delivered under chain-of-custody control to CBI of Gloucester, Virginia. This procedure will be repeated for three 24-hour sampling events within one week as required by 40CFR Part 136 WET testing protocols to supply water throughout the duration of the laboratory tests.

In addition to the 24-hour composite laboratory samples, the sample will be analyzed in the field for pH, total residual chlorine (TRC), and temperature analysis using calibrated meters. The pH and TRC analysis will be performed by Golder personnel who have completed the requisite IDC evaluation. The pH analysis will be performed using the Golder SOP for pH analysis (Attachment C), which conforms to EPA Method 4500 H+B. TRC analysis will be performed using the Golder SOP for TRC analysis (Attachment D), which conforms to EPA Method 8167.

A 24-Hour Composite Sampling Log will be used to record composite information, water quality measurements, and sample observations. Field observations will include visual monitoring of the presence of floating solids, visible foam, or sheen.

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### 2.2.4.2 Monthly WET Laboratory Analysis

The 24-hour composite samples will be analyzed by CBI, a VELAP-accredited laboratory, for the following WET analyses:

- EPA 1000.0: Chronic Toxicity Fathead Minnow, Pimephales promelas
- EPA 1002.0: Chronic Toxicity Daphnia, Ceriodaphnia dubia
- EPA 2000.0: Acute Toxicity Fathead Minnow, Pimephales promelas
- EPA 2002.0: Acute Toxicity Daphnia, Ceriodaphnia dubia

### 2.2.5 Backup Manual Sampling Procedure

One aliquot of equal volume will be collected each hour, for the monitoring period (4 or 24 total aliquots) directly from a grab sampling port in the effluent pipe exiting the storage tank into separate unpreserved plastic containers. The aliquot containers will be labelled and placed in a cooler on ice, for the duration of the 4-hour composite. Flow will be measured with a flow meter at the sampling location and recorded on a Composite Sampling Log (Attachment B). If flow is determined to be proportionate over the sampling interval (range within 10%), equal aliquots will be composited to represent a flow-proportioned sample. If the flow range is over 10%, Golder will calculate, measure, and composite flow-proportioned aliquots. Each aliquot will be calculated by multiplying the percentage of the aliquot's total flow by the total sample volume. A flow-proportioned aliquot will be measured using a decontaminated, graduated measuring cup and then placed in a decontaminated, lined carboy for compositing.

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### 3.0 FIELD PROCEDURES

This section summarizes Station field procedures for obtaining representative samples and analytical data for the 500-series outfalls. The procedures presented in this section have been developed to be consistent with EPA requirements.

### 3.1 Station Health and Safety Procedures

When conducting sampling activities, personnel will not enter the Station prior to coordinating with the Dominion Construction Manager, Alan Leatherwood and the Station ECCs (Randy Montaperto and/or Taylor Engen), and implementing additional safety measures if necessary. Required personal protective equipment (PPE) anticipated to ensure sampler safety includes Level D PPE in addition to Personal Flotation Devices (PFDs) for work over or near water. Golder personnel involved in sampling will don proper PPE prior to engaging in sampling activities.

### 3.2 Sampling Equipment

The following equipment will be used for effluent water sampling:

- Disposable nitrile sampling gloves
- Field notebook, sampling forms, chain-of-custody forms
- CorePro® tank sampler
- Disposable carboy liners
- 10-L Carboy
- ISCO 6712 Auto-sampler, or equivalent (composite samples only)
- Laboratory-provided, pre-labeled, pre-preserved sample containers
- Sample shipping container (cooler), with ice, as needed



### 3.3 Decontamination and IDW Handling

In general, sampling equipment will be disposable. If sampling equipment (*i.e.*, tank sampler) has direct sample contact, it shall be decontaminated prior to demobilization. Decontamination equipment and sampling equipment will be dedicated to the Bremo site and stationed at the Outfall 504 protective housing. Decontamination will require the following materials:

- Alconox®
- deionized water
- 5-gallon bucket(s) with tight fitting lids
- Scrub brush(es)

The procedure for decontamination will be as follows:

- 1. Wipe excess liquids or sediment from the equipment
- 2. Clean with Alconox® and deionized water; if needed, using a brush to remove obvious particulate matter and surface films
- 3. Rinse thoroughly with deionized water
- 4. Allow equipment to air dry

Investigation-derived wastes (IDW) will be managed as follows. Decontamination fluids, while expected to be minimal (less than 1 gallon per month), will be containerized and disposed of in accordance with Dominion's on-site instruction. Solid IDW, including gloves and disposable sampling equipment (e.g., tubing and carboy liners), will be disposed of as sanitary waste in a Station dumpster.

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### 4.0 FIELD DOCUMENTATION

Information pertinent to the sampling will be legibly recorded in bound and numbered field notebooks or on field forms (included in Attachment B) using indelible ink. Field notes should document:

- Date
- Project number
- Time of each data entry
- Description of work being performed that day
- Location on the Station
- Names and affiliations of personnel involved in sampling activities
- Weather conditions
- Calibration information, as needed
- Sample identification, methodology, and observations
- Other visual observations
- Pertinent field data, including measurements and meter readings (if not downloaded)
- Serial numbers on meters or other equipment
- Equipment calibration information
- Variations from procedures and documentation of Project Manager authorization
- Summary of phone calls concerning sampling

The spatial location for each sample will be documented with the general location sketch recorded in the field notes. This may include distance measurements (*e.g.*, depth and distance from the corner of a pond) or other indicators to allow the location to be identified later.

### 5.0 SAMPLE HANDLING

The laboratories will supply appropriately prepared sample containers and transportation containers (typically coolers) prior to each field event. Chemical preservatives may be needed for some analyses (e.g., metals analyses). In those cases, the laboratory will provide sampling containers pre-filled with the appropriate preservative type and amount for a particular analysis. Preservation procedures and analytical holding times will be in accordance with the published analytical methods.

Sample containers will be kept closed until the time they are to be filled. After filling, the containers will be securely closed and labeled with water-proof ink. The samples will be promptly placed in the cooler on ice.

Samples will be packed in a manner to minimize potential breakage of sample bottles. Packaging may entail the use of laboratory-supplied bubble wrap designed to fit the particular container. In general, samples will be shipped/delivered to the laboratory on the day of sample collection, or held for batch shipment if acceptable based on holding and turn-around times.

### 5.1 Chain-of-Custody

Samples submitted for laboratory analysis will be handled under chain-of-custody control beginning in the field. These standard industry-wide procedures have been established to ensure sample traceability from the time of collection through completion of analysis. For the purposes of this SAP, a sample is considered to be in a person's custody under the following conditions:

- 1. It is in one's possession, or
- 2. It is in one's view after being in one's possession, or
- 3. It was in one's possession and that person locked it up, or
- 4. It is in a designated secure area.

When custody of the samples is exchanged during field sampling, such transfer will be documented on a chain-of-custody form. The person relinquishing the samples will sign the form indicating the custody has been surrendered, and the receiving person will sign the form indicating an acceptance of responsibility for the custody of the samples. The chain-of-custody form will include:

- Sample identification number and matrix (*e.g.*, soil, water)
- Site name and project number
- Sampler's name or initials
- Sample collection date and time (i.e., military time)
- Designation as a grab or composite sample
- Requested analyses
- Any chemical preservatives



Ref. 15-20347.220 June 2016

- Turn-around time for sample analytical results
- pH results and grab sample time (for weekly samples)
- Any special comments

When transferring the samples directly to the laboratory, such as to their courier, one member of the sampling team will sign the chain-of-custody form relinquishing custody to the laboratory. The receiving laboratory representative will also sign the form. The sampling team will retain a copy for the project files.

If using an overnight courier service (e.g., FedEx) to ship the samples to the laboratory, the form will be signed by a sampling team member, the tracking number for the shipping label(s) will be recorded on the form, and the original form will be placed inside the shipping container with the samples. The form will be enclosed in a plastic bag to prevent water damage to the paperwork. A copy of the form will be retained for the files. The overnight courier does not need to sign the form.

The shipping container will be securely closed (*e.g.*, with packing tape). When a non-laboratory courier or other non-Golder person (shipping company) is given custody of the samples, the shipping container will be sealed with a signed and dated custody seal.

Once samples are transported to the laboratory, custodial responsibility is transferred to the laboratory's sample manager (or similar position) to assure that the procedures presented in the laboratory's Quality Assurance Plan (QAP) and the appropriate analytical methods are followed. The laboratory QAP will contain a detailed description of the laboratory chain-of-custody procedures, including receipt of samples, designation of a sample custodian, custody within the laboratory, and laboratory storage and disposal procedures.

### 6.0 DELIVERABLES

The following sections discuss data quality assurance / quality control (QA/QC) and deliverables:

### 6.1 Data QA/QC

Golder will contract with the laboratories to ensure that TATs and data quality objectives are met. Golder will receive laboratory results within 24 hours of sample receipt at the laboratory. Upon receiving analytical results, Golder will enter the electronic data deliverables (EDDs) into EQUiS, a data management tool. Golder will use EQUiS to check applicable QLs, assure that the parameters required by the permit are reported, and compare results to the permitted limits. If data quality objectives are not met, Golder will coordinate with the laboratory to remedy the errors.

### 6.2 24-Hour Exceedance DEQ Notification

The VPDES permit requires 24-hour DEQ notification of permit limit exceedances, per Section I.G.22. Within 24 hours of receipt of each data package, Golder will provide a pass, fail, or QC status update to Dominion, with final data packages and a permit limit comparison for each weekly sampling event via electronic mail in a format acceptable to Dominion.

### 6.3 Weekly Sampling Summary

The VPDES permit (Section I.9.A.h) requires weekly summaries to be submitted to DEQ. Golder will prepare a sampling activities letter and a tabulated data summary for the three weekly 500-series samples for Dominion's review by Wednesday of the following week.

### 6.4 Monthly Discharge Monitoring Report

The VPDES permit requires monthly Discharge Monitoring Report (DMR) submittals by the 10<sup>th</sup> of each month. Golder understands that Dominion submits electronic DMRs (eDMRs) to DEQ. To facilitate Dominion's production of eDMRs, Golder will provide final monthly WET analysis data packages, permit limit comparisons for each monthly sampling event, and statistical analysis. Statistical evaluations will include daily maximum, daily minimum, weekly average, and monthly average. Golder will prepare a tabulated data summary for the 500-series outfalls that will mimic the eDMR format, and submit the report to Dominion via electronic mail by the 5<sup>th</sup> of each month.



### **Attachment A**

VPDES Permit VA0004138 Section I.A.9

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# A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS

During the period beginning with the permit's effective date and lasting until completion of dewatering activities, or until the permit's expiration date, whichever comes first, the permittee is authorized to discharge from internal Outfall 501 (process wastewater from dewatering activities in the West Ash Pond), Outfall 502 (process wastewater from dewatering activities in the East Ash Ponds), Outfall 504 (combination of process wastewaters from dewatering activities in the North Ash Ponds), Outfall 504 (combination of process wastewaters from dewatering activities in the West Ash Pond. North Ash Pond, East Ash Ponds, and Metals Cleaning Waste Treatment Basin), and Outfall 505 (process wastewater from dewatering activities in the Metal Cleaning Waste Treatment Basin). Any process wastewater removed from the West Ash Pond. North Ash Pond, and East Ash Ponds for discharge purposes is considered to be process wastewater from dewatering activities. See Part I.G.19 for requirements regarding the decanting and dewatering of the Metal Cleaning Waste Treatment Basin.

This discharge shall be limited and monitored as specified below:

EFFLUENT CHARACTERISTICS	DISCH/	DISCHARGE LIMITATIONS			MONITORING REQUIREMENT	EQUIREMENTS
	Monthly Average	Weekly Average	Minimum	Maximum	Frequency	Sample Type
Flow (MGD) a	N	ΥZ	NA	Ŋ	1/Day	Estimate
pH (standard units) <sup>h,j</sup>	NA A	NA	0.9	0.6	3/Week	Grab
Total Suspended Solids (mg/L) b.h.j	30.0	ΥZ	NA	100.0	3/Week	4 HC
Oil & Grease (mg/L) <sup>b.h.j</sup>	15.0	NA	NA	20.0	3/Week	4 HC
Total Recoverable Antimony (ug/L) b.h.j	2,100	NA	NA A	2,100	3/Week	4 HC
Total Recoverable Arsenic (ug/L) <sup>b,h,j</sup>	290	NA AN	NA	530	3/Week	4 HC
Total Recoverable Cadmium (ug/L) bhj	1.8	N.A.	NA	3.2	3/Week	4 HC
Total Recoverable Chromium III (ug/L) bhj	120	NA	NA	220	3/Week	4 HC
Total Recoverable Chromium VI (ug/L) bhj	81	NA	NA	34	3/Week	4 HC
Total Recoverable Copper (ug/L) bhi	12	NA	NA A	23	3/Week	4 HC
Total Recoverable Lead ( $ug/L$ ) $^{b,h,j}$	61	N.A.	NA	35	3/Week	4 HC
Total Recoverable Mercury (ug/L) bhj	1.5	NA	NA	2.8	3/Week	4 HC
Total Recoverable Nickel (ug/L) bhj	31	NA	NA	57	3/Week	4 HC
Total Recoverable Selenium (ug/L) <sup>b.h.j</sup>	9.6	N.A.	ΑN	18	3/Week	4 HC
Total Recoverable Silver (ug/L) bhj	2.7	NA AN	N.A.	5.0	3/Week	4 HC
Total Recoverable Thallium (ug/L) bhj	1.4	NA	Ϋ́	1.4	3/Week	4 HC
Total Recoverable Zinc (ug/L) bhj	110	NA	Ϋ́Α	210	3/Week	4 HC
Chloride (mg/L) <sup>b.h.j</sup>	450	A'N	NA A	820	3/Week	4 HC
Ammonia-N (mg/L) <sup>b.h.j</sup>	9.6	NA A	ΑN	14	3/Week	4 HC
Hardness (mg/L as CaCO <sub>2</sub> ) hJ	N	NA A	NA	Z	3/Week	4 HC
Cyanide, Free (ug/L) <sup>i</sup>	Z	NA	ΥN	N	1/Month	4 HC
Total Recoverable Aluminum (ug/L) i	N	ΝΑ	Ϋ́Ζ	Ŋ.	1/Month	4 HC
Total Recoverable Barium (ug/L) i	N	ΝΑ	NA	Ä	1/Month	4 HC
Total Recoverable Beryllium (ug/L)	N	NA	ΝΑ	Ä	1/Month	4 HC
Total Recoverable Boron (ug/L)	Z	ΝΑ	NA	Ŋ	1/Month	4 HC
Total Recoverable Cobalt (ug/L) i	N	NA	NA	Ŋ	1/Month	4 HC

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EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS Ą

9. Continued from previous page

EFFLUENT CHARACTERISTICS	DISCHA	DISCHARGE LIMITATIONS			MONITORING R	IONITORING REQUIREMENTS
	Monthly Average	Weekly Average	Minimum	Maximum	Frequency	Sample Type
Total Recoverable Iron (ug/L)	ď	ZA	Ϋ́	Z	1/Month	4 HC
Total Recoverable Molybdenum (ug/L)	Ŋ	NA	NA	Z	1/Month	4 HC
Total Recoverable Vanadium (ug/L)	Ŋ	NA A	NA	Ŋ	1/Month	4 HC
Acute Whole Effluent Toxicity, Ceriodaphnia dubia (%) c.j	NA	AN	100	NA	1/Month	24 HC
Chronic Whole Effluent Toxicity, Ceriodaphnia dubia (TUc) ci	NA	NA	NA A	6.25	1/Month	24 HC
Acute Whole Effluent Toxicity, Pimephales promelas (%) c,i	NA	NA	100	NA	1/Month	24 HC
Chronic Whole Effluent Toxicity, Pimephales promelas (TUc) <sup>e,j</sup>	Ϋ́Ζ	NA	NA	6.25	1/Month	24 HC

4 HC = 4- hour Composite NA = Not ApplicableNL = No Limitation, monitoring required

24 HC = 24- hour Composite

See Part I.C for additional monitoring instructions. The limits are based on a flow of 10.2912 MGD.

a .c.

See Part I.E for additional monitoring instructions.
The discharges from internal Outfalls 501, 502, 503, 504, and 505 are authorized to discharge to the Stormwater Management Pond and West Treatment Pond and through Outfalls 002, 003, 004, and/or 006. رت ت

Ash dewatering water (pore water within the coal combustion residuals mass) and contact stormwater (stormwater that has contacted the coal combustion residuals) are process wastewater from dewatering activities.

Compliance with the limits above may be demonstrated with or without additional treatment.

There shall be no discharge of floating solids or visible foam in other than trace amounts. عرہ نب

Sampling for the parameters identified with a monitoring frequency of "3/Week" for Internal Outfalls 501, 502, 503, 504, and 505 shall occur at least three (3) days per week with a minimum of 48 hours between sampling events. A sampling week extends Sunday through Saturday. The permittee shall contract to receive results for parameters identified business Friday of the week following sample collection. This reporting requirement does not substitute for, or alter, Part II.C concerning the monthly reporting of monitoring with a monitoring frequency of "3/Week" within four business days of taking the sample. Results of the weekly sampling shall be reported to DEQ no later than the close of results with the Discharge Monitoring Report.

The composite period for the parameters identified with a monitoring frequency of "1/Month" for Internal Outfalls 501, 502, 503, and 505 shall occur within the composite period for the Whole Effluent Toxicity monitoring.

See Part I.G.22 for additional requirements.

### **Attachment B**

Sampling Logs



### **Tank Composite Sampling Log**

Golder							D	ate:			
Associate	S						W	eather	:		
te:_Bremo Power Stat	ion	Permi	it Number:_VA	.0004138	8						
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boratory Sample Co	llection:	Sample ID:					Samp	le Tim	e:		
			Sample Obs	ervations	S						
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Odor	□ None	☐ Othe									
Clarity	☐ Clear		ntly cloudy	☐ Clou	ıdv	☐ Opaqu					
Floating Solids	None		(Describe):	<u> </u>	idy	<u> </u>					-
Foam	None		(Describe):								
Sheen	None	☐ Bloci	<u> </u>	☐ Swirl	ıls						
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eld Analysis:    Oakton PCTestr35 (   Analyst has complet     PH meter has been (   Result: _   rbidity: HACH 21000     Ioride: HACH 8-P(#A     Titrant Lot (exp): A6     Drops: _   nmonia-N: LaMotte (exp)     Reagent #1 Lot (exp)     Reagent #1 Lot (exp)     Reagent #2 Lot (exp)     Reagent #4 L	ed an initial calibrated in SU  (#45375)  Result:  6134) A 6125 (05/18)  Colorimeter p): 485218  sult: cont results be	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Reamg/L elow QL 0.05 m	ation of capabilities with EPA M  C Resorated:  J Result    E: Ser Lot (exp): A6  mg/L    onia-N (#2760 agent #2 Lot (exp): A6  g/L as < 0.05	Lity for persent standard (076 (03/2 Result I 03-0916) exp): 096	erforming the following states of the following states	ing pH and B (21st extends limits  Time: rrelation  It: 00mg/L S permit limits  Time: vertical permit limits  yether permit limits	alysis? dition)' (6.0 limit (4mg/L tandar mit (82	9.0 SI  5 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Tes	/2′ <b>N</b> o
eld Analysis:  1: Oakton PCTestr35 ( Analyst has complet pH meter has been of Result:	ed an initial calibrated in SU  (#45375)  Result: .6134) A 6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Result lelow QL 0.05 m Colorimeter I	ation of capabilities with EPA M  C Resorated:  J Result    Result    E:	Lity for percent of the lethod 45 sult with Anabelow To Standard (076 (03/2 Result In 193-0916) exp): 096 Result Time Zee	erforming to the serious of the seri	ing pH and B (21st extends a B) Time: Trelation It: 00mg/L S permit line Zeroed: (4/18) v permit I	alysis? dition)' (6.0 – limit (4 _ mg/L tandar mit (82	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/21 <b>N</b> c
eld Analysis:  L: Oakton PCTestr35 ( Analyst has complet pH meter has been of Result:	ed an initial calibrated in SU  (#45375)  Result: .6134) A 6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Reamg/L elow QL 0.05 m	ation of capabil the with EPA M  C Res  Practed: T Result  Result  A Result	lity for per lethod 45 sult with An below To Standard 1076 (03/2 Result In 103-0916) exp): 096 Result Time Zee	erforming to the serious of the seri	ing pH and B (21st extends limits  Time: rrelation  It: 00mg/L S permit limits  Time: vertical permit limits  yether permit limits	alysis? dition)' (6.0 – limit (4 _ mg/L tandar mit (82	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/21 <b>N</b> c
eld Analysis:  Delta Del	ed an initial calibrated in SU  (#45375)  Result: .6134) A 6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Result lelow QL 0.05 m Colorimeter I	ation of capabilities with EPA M  _ °C	lity for per lethod 45 sult with Anabelow To Standard 1076 (03/2 Result In 103-0916) exp): 096 Result Time Zee	erforming to the serious of the seri	ing pH and B (21st extends a B) Time: Trelation It: 00mg/L S permit lim the Zeroed: (4/18) v permit I	alysis? dition)' (6.0 – limit (4 _ mg/L tandar mit (82	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/2′ <b>N</b> o
eld Analysis:  : Oakton PCTestr35 (     Analyst has complet pH meter has been of Result:	ed an initial calibrated in SU  (#45375)  Result: .6134) A 6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18 .6125 (05/18	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Result lelow QL 0.05 m Colorimeter I	ation of capabilities with EPA M  C Resorated:  J Result  E: S  T Lot (exp): A6  mg/L  onia-N (#2760 agent #2 Lot (exp): A6  g/L as < 0.05  I (#292691)  Result  C 2 mg/L  C 5 mg/L  C 5 mg/L	lity for per lethod 45 sult with  Anabelow To Standard 1076 (03/2)  Result In 103-0916) (exp): 096	erforming to the serious of the seri	ing pH and B (21st extends a limit limits) Time: Trelation It: 00mg/L S permit limits Depreciation The Zeroed: (4/18) The Permit limits Time: The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18)	alysis? dition)' (6.0 limit (4 _ mg/L tandar mit (82 imit (1	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/2′ <b>N</b> o
Id Analysis: Coakton PCTestr35 ( Analyst has complet pH meter has been of Result: Thidity: HACH 2100C  Ioride: HACH 8-P(#A Titrant Lot (exp): Ac Drops: Monia-N: LaMotte C Reagent #1 Lot (ex Preprint Proposed Pr	ed an initial calibrated in SU  (#45375)  Result: 6134) A 6125 (05/18  Colorimeter p): 485218 sult: cont results be control of the colorimeter be colorimeter by colorimeter be colorimeter by colorimeter be colorimeter by colorimete	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Result lelow QL 0.05 m Colorimeter I	ation of capabilities with EPA M  "C Resorated:  J Result    e:	lity for per lethod 45 sult with  Anabelow To Standard 1076 (03/2)  Result In 103-0916) (exp): 096	erforming to the serious of the seri	ing pH and B (21st extends a limit limits) Time: Trelation It: 00mg/L S permit limits Depreciation The Zeroed: (4/18) The Permit limits Time: The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18)	alysis? dition)' (6.0 - limit (4 _ mg/L tandar mit (82  imit (1	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/2 <sup>-</sup> /2
Id Analysis: Coakton PCTestr35 ( Analyst has completed pH meter has been of Result: Thidity: HACH 2100C  Ioride: HACH 8-P(#A Titrant Lot (exp): AC Drops: Commonia-N: LaMotte Coagent #1 Lot (exp): Reagent #1 Lot (exp): Cuvette ID  Sample  Cal 1 (2 mg/L)	ed an initial calibrated in SU  (#45375)  Result:  .6134) A .6125 (05/18  Colorimeter p): 485218  sult: .oort results be .CH TPH C	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Result lelow QL 0.05 m Colorimeter I	ation of capabilities with EPA M  "C Resorated:  J Result    Be:	lity for per lethod 45 sult with  Anabelow To Standard 1076 (03/2)  Result In 103-0916) (exp): 096	erforming to the serious of the seri	ing pH and B (21st extends a limit limits) Time: Trelation It: 00mg/L S permit limits Depreciation The Zeroed: (4/18) The Permit limits Time: The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18)	alysis? dition)' (6.0 limit (4 _ mg/L tandar mit (82 imit (1 - ermit li ] Yes ] No N/A	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/2 <sup>-</sup> /2
eld Analysis:  : Oakton PCTestr35 (     Analyst has complet pH meter has been of Result:	ed an initial calibrated in SU  (#45375)  Result:  .6134) A .6125 (05/18  Colorimeter p): 485218  sult: .oort results be .CH TPH C	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Result lelow QL 0.05 m Colorimeter I	ation of capabilities with EPA M  "C Resorated:  J Result  e:	lity for per lethod 45 sult with  Anabelow To Standard 1076 (03/2)  Result In 103-0916) (exp): 096	erforming to the serious of the seri	ing pH and B (21st extends a limit limits) Time: Trelation It: 00mg/L S permit limits Depreciation The Zeroed: (4/18) The Permit limits Time: The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18)	alysis? dition)' (6.0 - limit (4 _ mg/L tandar mit (82  imit (1	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/2′ <b>N</b> o
eld Analysis:  Analyst has complet pH meter has been of Result:  Inbidity: HACH 2100C  Iloride: HACH 8-P(#A Titrant Lot (exp): AC Drops:  Inmonia-N: LaMotte C Reagent #1 Lot (ex *rep.)  I & Grease (TPH): HA Reagent Lot (exp):  Cuvette ID  Sample  Cal 1 (2 mg/L)	ed an initial calibrated in SU  (#45375)  Result:  .6134) A .6125 (05/18  Colorimeter p): 485218 sult: .cort results be .CH TPH C	al demonstra n accordance at Time CalibNTU analysis Time 8); Indicator Result: r 1200 Amm 8 (1/19); Result lelow QL 0.05 m Colorimeter I	ation of capabilities with EPA M  "C Resorated:  J Result    Be:	lity for per lethod 45 sult with  Anabelow To Standard 1076 (03/2)  Result In 103-0916) (exp): 096	erforming to the serious of the seri	ing pH and B (21st extends a limit limits) Time: Trelation It: 00mg/L S permit limits Depreciation The Zeroed: (4/18) The Permit limits Time: The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18) The Depreciation of the limits The Zeroed: (4/18)	alysis? dition)' (6.0 limit (4 _ mg/L tandar mit (82 imit (1 - ermit li ] Yes ] No N/A	9.0 SI  15 NTL  d Lot ( 0 mg/l	s	Yes  A6110 (4 Yes  Yes  Yes  Yes	/21 <b>N</b> o



Associates    Date:	
Permit Number:_VA0004138 Outfall Number:_504_  Aliquot Collection and Compositing:  Equipment: ISCO 6712 Auto-Sampler (SN 215K02095), Pressurized line sampler, 10L carboy, HACH 2100Q (45375)	
Aliquot Collection and Compositing:  Equipment: ISCO 6712 Auto-Sampler (SN 215K02095), Pressurized line sampler, 10L carboy, HACH 2100Q (45375)	
Equipment: ISCO 6712 Auto-Sampler (SN 215K02095), Pressurized line sampler, 10L carboy, HACH 2100Q (45375)	
Flow-paced Sample Collection Program:	5)
1. Select PROGRAM and Site Description Change NO 2. Number of Bottles: 1 3. Bottle Volume: 10,000 mL 4. Suction Line Length: 10 FT 5. FLOW PACED 6. Flow between sample events: 0.026 Mgal 7. BOTTLES/SAMPLES  8. Run Continuously?: NO 9. Bottles per Sample Event: 1 10. CLOCK TIME 11. Start Flow Count at: 60:00 12. Select Days: □SU □MO □TU □WE □TH □FR 13. Programming Complete Run this Program Now?	
Sample Data:	
Start Time: End Time: Total sample collected: L Aliquot Amount: m	ıL
Turbidity Meter Calibrated Date/Time: Composite Sample Turbidity: N	10
Laboratory Sample Collection:	
Sample ID:Outfall 504 Sample Time:	
Sample Observations:	
Color	
Odor Other:	
Clarity	
Floating Solids    None    Yes (Describe):  Foam    None    Yes (Describe):	
Foam   □ None   □ Yes (Describe):   Sheen   □ None   □ Blocky   □ Swirls	
Analysis: □ TSS; Oil & Grease; Metals: Sb, As, Cd, Cr(III), Cr(VI), Cu, Pb, Hg, Ni, Se, Ag, Th, and Zn; Chloride; Ammonia-N (3 □ Free Cyanide; Metals: Al; Ba; Be; Bo; Co, Fe, Mo, and V (1/mo) □ Other:	3/wk)
pH Calibration/Certification:	
Instrument Make/Model: _Oakton PCTestr35_ Serial No: _2433781_ Date/Time Calibrated:	_
Analyst has completed an initial demonstration of capability for performing pH analysis? ☐ Yes ☐ No pH meter has been calibrated in accordance with EPA Method 4500-H+ B (21st edition)? ☐ Yes ☐ No	
pH Analysis:	
Sample Collection Time: Sample Analysis Time: pH Sample Result: At:(°C)	

Notes: pH calibrations are performed prior to sampling and at the beginning of each day of sampling. The temperature compensating thermometer (thermistor) for the pH meter is verified annually in accordance with SM 2550 B.1



### 4-Hour Manual Composite Sampling Log

	Assucia	ics				Masthan		
a		<b>6</b> . ()				vveatner:		_
	ation:_Bremo P							
	Number:_VA000					Outfall Num	nber:_504_	
<u>Aliquot</u>	Collection and	Compositing:						
	AP. AN.	Time Collected	Amount	Flow	Percent of	Flow-Prop	ortioned Aliquot*	1
	Aliquot No.	(1/hr)	Collected	Rate	Total Flow		ollected)x(% Flow)	
	1							4
	2							4
	3 4							1
	Total							1
*If range b	etween maximum flo	ow and minimum flow is	>10%, flow-prop	ortioned a	I liquots must be c	alculated, measur	ed, and composited.	
-								
Equipme	ent: _1-L aliquot	bottles, measuring	cup, lined car	rboy				
Sample	Collection Method	ods: _Collect 2 1-L	bottles aliquo	ts from (	grab sample p	ort, collect flov	v during aliquot colle	∍ction,
calculate	e flow-proportion	ned composite, mea	asure represer	ntative a	liquots, comp	osite flow-prop	ortioned sample	
Laborat	ory Sample Co	llection:						
	ID:Outfall 50	4 Sample Tir	me:	<del></del>				
Sample	Observations:							
	Color		☐ Other:					
	Odor	☐ None	Other:					
	Clarity	☐ Clear	Slightly clo	udy	☐ Cloudy	□ Opaque		
	Floating So	lids 🔲 None	Yes (Descr	ribe):				
	Foam	☐ None	Yes (Descr	ribe):				
	Sheen	☐ None	□ Blocky		☐ Swirls			
☐ Free	Oil & Grease; M Cyanide; Metals	: Al; Ba; Be; Bo; Co	, Fe, Mo, and			g, Th, and Zn;	Chloride; Ammonia-	-N (3/wk
Other	•				_			
pH Cali	oration/Certifica	ation:						
l m mtm, mm	ont Maka/Madali	Ookton DCTootra	E Carial N	la: 24	22701 Doto	Timo Calibrata	.d.	
				10:24	33781Date	Time Calibrate	ed:	
Date In	ermistor Last Cr	necked:factory	-					
		an initial demonstrat						
pH mete	er has been calib	rated in accordanc	e with EPA M	ethod 48	500-H+ B (21°	tedition)? 🔲 ۱	∕es 🔲 No	
pH Anal	lysis:							
Sample	Collection Time:	: Sample <i>i</i>	Analysis Time	e:	pH Sample	Result:	At: (°C)	
-1			•	•				
Eiold An	alveis Dorformo	d Rv:		Data.				

Notes: pH calibrations are performed prior to sampling and at the beginning of each day of sampling. The temperature compensating thermometer (thermistor) for the pH meter is verified annually in accordance with SM 2550 B.1

Page 1 of 1

### 24-Hour Composite Auto-Sampling Log

V	Associates					Date:	
						Veather:	
Sit	e Location:_Bremo Power Station	on			5	Sampler:	
Permit Number:_VA0004138					Outfall Number:_504_		
<u>AI</u>	iquot Collection and Composi	ting:					
Ε¢	uipment: ISCO 6712 Auto-Sam	pler (215K020	95), Pressuri	zed line	e sampler, 10l	_ carboy, disposable li	iner
Fle	ow-paced Sample Collection P	rogram:					
1.	Select PROGRAM and Site De	elect PROGRAM and Site Description Change NO 8. Run Continuously?: NO				ısly?: NO	
2.	Number of Bottles: 1			9. B	ottles per Sar	mple Event: 1	
3.	Bottle Volume: 10,000 mL				10. CLOCK TIME		
	Suction Line Length: 10 FT				tart Flow Cou	nt at: 60:00	
5.						JSU DMO DTU DWE	E DTH DER DSA
_		0.006 Maal			•		
6. 7.	Flow between sample events: BOTTLES/SAMPLES	0.026 Nigai		13. F	rogramming (	Complete Run this Pro	ogram Now?. NO
	I <b>mple Data:</b> Imple ID:	Start Da	ate/Time:			End Date/Time:	
То	tal sample collected: L						
<u>La</u>	boratory Sample Collection:						
Sa	imple Observations:						
	Color	☐ None	☐ Other:				]
	Odor	□ None	Other:	I = <b></b> .	T Claudy		
	Clarity Floating Solids	☐ Clear☐ None	☐ Slightly c☐ Yes (Des		│ □ Cloudy	☐ Opaque	
	Foam	None	☐ Yes (Des				
	Sheen	☐ None	☐ Blocky		☐ Swirls		
	alysis: EPA Methods 1000.0, 1002.0, 2	2.5 gallon (Da	ay 2) 🔲 4	gallon ( ——	Day 3)		
Ins	RC Calibration/Certification: etrument Make/Model: _HACH P piration of Spec✓™ gel standard ate/Time Calibration Verified:	ds: _03/2017_			ial No: _A534 iration of DPD	9_ ) Total Chlorine Reag	ent: _12/2020
HA	nalyst has completed an initial de ACH Pocket Colorimeter II has b						
Sa	RC Analysis: Imple Collection Time: Imple Read Time (within 3-6 min	Reagen utes of reager	nt Additive Tin	ne: ne):	т	RC Sample Result: _	mg/L
Fie	eld Analysis Performed By:		Date	):			Page 1 of 2



### 24-Hour Composite Auto-Sampling Log

pH Calibration/Certification: Instrument Make/Model: _Oakton PCTestr35_	No: _2433781 Date/Time Calibrated	:					
Analyst has completed an initial demonstration of capability for performing pH analysis? ☐ Yes ☐ No pH meter has been calibrated in accordance with EPA Method 4500-H+ B (21st edition)? ☐ Yes ☐ No							
pH Analysis: Sample Collection Time: Sample Analysis Time	e: pH Sample Result:	At:(°C)					
Notes: pH calibrations are performed prior to sampling and at the the (thermistor) for the pH meter is verified annually in accordance with SI	peginning of each day of sampling. The tempera M 2550 B.1	ture compensating thermometer					
Field Analysis Performed By:	Date:	Page 2 of 2					

### **Attachment C**

pH Standard Operating Procedure



### Standard Operating Procedure – pH Analysis

### **VPDES PH ANALYSIS** 1.0

This standard operating procedure (SOP) is for the pH analysis of treated water at the Bremo Power Station required by the Virginia Pollutant Discharge Elimination System (VPDES) Permit. The following procedures are based on the revised 2011 online edition of Environmental Protection Agency (EPA) Standard Method (SM) 4500-H+ B [pH (electrometric) Monitoring]. The pH meter used for analysis at Bremo is an Oakton PCTestr35.

### 2.0 **CARE AND MAINTENANCE**

The condition of the pH meter and calibration buffer solutions will be inspected prior to performing pH meter calibration described in the subsequent sections of this document. The following items will be inspected, documented, and corrected prior to pH meter calibration.

### 2.1 pH Meter

- The glass pH electrode should be free of scratches, staining, or buildup of precipitate. Clean the electrode with a chemical wipe or replace the electrode if conditions effect meter stabilization during calibration.
- Check to make sure the electrode is no older than 1 year.
- Perform a calibration/check to the pH meter thermometer (thermistor) annually using EPA SM 2550 B.1 and procedure outlined in Section 2.3.

### 2.2 Calibration Buffer Solutions

- Confirm the buffer solutions are traceable to National Institute of Technology (NIST)
- Store buffer solution according to manufacturer's instructions.
- Check shelf life of buffer solutions and replace if necessary.
- Check the buffer solutions for contamination or organic growths, and replace if necessary.

Document any changes or repairs to the pH meter in the notes field of the pH field calibration log presented in Attachment I.

### 2.3 pH Meter Thermistor Annual Check

The pH meter thermistor will be checked or verified annually against a thermometer that has been certified and calibrated against NIST approved standards in accordance with EPA SM 2550 B.1. The following procedures are for the annual verification of the pH meter:

- 1. Collect 200 milliliters (ml) of tap water in a 250 ml graduated cylinder.
- Place the NIST traceable thermometer pH meter electrode in the water.
- Allow for the two thermometers to stabilize  $(+/-0.1^{\circ}C)$ .
- The two stabilized readings should be within +/-0.1°C or the NIST thermometer's certified correction chart. Follow manufacturer's recommendations or replace it the pH meter, if the thermistor is unable to stabilize under this criterion.
- Record the following on the annual thermometer verification log presented in Attachment 11:
  - Make and model numbers of the NIST traceable thermometer and the pH meter.





### Standard Operating Procedure – pH Analysis

- Date and time of the verification.
- The temperature of the water measured by the NIST traceable thermometer and the pH
- The analyst's name.

### INITIAL DEMONSTRATION OF CAPABILITY 3.0

The initial demonstration of capability (IDC) will be performed once by the analyst/operator to ensure acceptable precision, accuracy, and sensitivity are provided for pH analysis. The IDC uses the revised 2011 online edition of EPA Standard Method 4500-H+ B as a reference and will be performed by each analyst/operator prior to performing a pH analysis. The IDC will be documented on the IDC form presented in Attachment III and performed in the presence of another analyst/operator who has completed the IDC, as a means quality control, using the following procedures:

- 1. Perform a three point calibration for pH analysis using the method in Section 4.0.
- 2. Analyze 4 additional buffer solutions of a different lot number from those used for the pH calibration. The recovery for each of the 4 samples must be +/- 0.1 Standard Units (S.U.) of the known concentration of the sample to be acceptable.
- Record the following on the IDC form:
  - Name of analyst performing the IDC;
  - Date of the IDC:
  - pH meter make, model and serial number;
  - Confirm that the pH meter's automatic temperature correction is enabled;
  - Confirm the internal thermometer has been calibrated/checked annually;
  - pH buffer solution standard for the calibration and reference samples;
  - pH buffer solution lot numbers for the calibration and reference samples;
  - Expiration date of pH buffer solution for the calibration and reference samples;
  - Calibrated temperature and pH for each buffer solution;
  - Reference sample temperature and pH for each buffer solution;
  - and, confirm that the IDC was acceptable by the quality control analyst.

A copy of the analyst IDC form will be maintained in the Bremo Field Sampling calibration binder.

### PH METER CALIBRATION 4.0

The pH meter will be calibrated daily for each day it is used for analysis. The pH meter will be calibrated according to the manufacturer recommendations and the procedures described below. A pH calibration log will be used to document the calibration. Instructions for the Oakton PCTestr35 calibration are presented in Attachment I. The following procedures are for calibrating the pH meter in accordance with the revised 2011 online edition of EPA SM 4500-H+ B.

- The analyst will don disposable nitrile gloves for the duration of the calibration.
- Fill four 250 milliliter (ml) graduated cylinders with 200 ml of pH calibration buffer solutions 7, 4, 10, and a duplicate sample composed of a second buffer 7 solution with a different lot number from the first. Allow the temperatures of the solutions to stabilize within +/- 1 degree.
- Calibrate the pH meter according to manufacturer instructions and these procedures:





### Standard Operating Procedure – pH Analysis

- Rinse the pH meter electrode with de-ionized (D.I.) water and blot dry with chemical wipes between buffer solutions during the calibration.
- Stir the pH meter electrode gentle at a constant speed in the buffer solution while calibrating.
- The duplicate sample will be analyzed once the calibration is completed for 7, 4, and 10 and must be +/- 0.1 S.U. of the original sample. Re-inspect the pH meter electrode for damage, re-calibrate the meter, and follow manufacturer instructions if the duplicate is not +/- 0.1 S.U.
- 4. Record the following on the pH field calibration log.
  - Date and time of the calibration;
  - pH buffer solution standard;
  - pH buffer solution lot number;
  - Expiration date of pH buffer solution;
  - Temperature of pH buffer solution when calibrated;
  - Calibrated reading saved to the pH meter;
  - Duplicate pH reading and temperature;
  - and, the analyst name that performed the calibration.

### 5.0 PH ANALYSIS

Once the pH meter has been calibrated in accordance with Section 4.0 of this document, it can be used for sample analysis at the series 500 Outfalls. The following is a list of procedures for collecting and analyzing effluent samples for pH analysis.

- Purge the effluent sample tap located in the effluent line according Sampling and Analysis Plan.
- Collect a 100 mL sample of effluent water in a graduated cylinder and place the pH meter electrode in the sample.
- Record the sample collection time on the Composite Sampling Log.
- Stir the sample gently at a constant speed while the pH meter stabilizes.
- Record the pH and temperature reading and the time the readings were collected on the field monitoring log once the pH has stabilized (+/- 0.1 S.U.). The sample must be analyzed within 15 minutes of the sample collection time.

### **Attachments**

Attachment I: pH Calibration Log Attachment II: Thermometer Calibration

Attachment III: Initial Demonstration of Capability

Attachment IV: Oakton PCTestr35 Calibration Instructions



G:Projects/IBM 0839-6351/VPDES Permit/March 2013 Va DEQ Inspection/Response to DEQAttachment Bl-Attachment II - pH Calibration Sheet 05-02-2013.xisx

		Analyst Name													
	Notes:	pH Probe Condition, Equipment Maintenance, Probe Replacement, etc.													
		Date Thermistor Last Checked <sup>3</sup>													
ion Log	Duplicate Analysis <sup>2</sup>	U													
pH Field Calibration Log	Duplicat	Hd										-			
	Reading	Temp °C													
Serial Number:	Calibrated Reading	Нd													
1		Expiration Date													
		Lot Number					-								
		pH Standard													
el number: _		Time													
Make Type/ Model number:		Date													

Notes: 1. pH calibrations must be performed in accordance with the EPA Approved On-Line Edition of Standard Method (SM) 4500.H\* B-00;
2. Only one duplicate analysis of a pH standard (typically 7.0) required per event, duplicate must be within 0.1 pH standard unit of calibrated reading; and
3. The temperature compensating thermometer (thermistor) for the pH meter must be verified annually in accordance with EPA SM 2550 B.1.

### **Annual Thermometer Verification Log**

acility Name:
lational Institute of Standards and Technology (NIST) Calibrated Thermometer:
lake Type/ Model number:
erial Number:
hermometer being verified:
lake Type/ Model number:
erial Number:

Date	Time	NIST Thermometer Temp °C	Thermometer Temp °C	Analyst Name

Notes: Verification performed in accordance with EPA Standard Method 2550 B.1



### **INITIAL DEMONSTRATION OF CAPABILITY**

EPA Standard M	ethod:	4500-	H <sup>⁺</sup> B [pH (elect	rome	tric)	Monitoring]	
Demonstration D	Pate:						- Andrew Control of the Control of t
Analyst/Operato	<b>r:</b>						
Matrix:		Water					
Monitoring Devi	ce:						
Is Meter Automa Has the thermor	-						
Calibration:							
Standard Units	Lot No. Sol	ution	Expiration Da	ate	Cali	brated pH	Temp. °C
	the fourth stan of number from he known cond Lot No.	Calibra	ation Standards on of the sampl <b>Expiration</b>	. Red			es/buffers must be ne 4 samples must Recovery
	Solution		Date		-		acceptable? Y/N
Demonstration a	·						
Analyst/Operato	r Signature:						
Quality Control	Officer Signati	ıre:					
Referenced EPA	Standard Met	hod 45	500-H <sup>+</sup> B 21 <sup>st</sup> eo	dition	1		



### **MULTI-PARAMETER TESTR 35 SERIES** pH / Conductivity / TDS / Salinity / Temperature INSTRUCTION MANUAL

### OAKION MCTEOT:



68X441601 Rev. 1 March 2010 Part of Thermo Fisher Scientific

Thank you for selecting our Multi-Parameter Testr. This manual serves the following (3) models: PCTestr 35 (Eutech PCTEST35-01X441504 / Oakton 35425-00) pH / Conductivity / Temperature

- PTTestr 35 (Eutech PCTEST35-01X441505 / Oakton 35425-05) pH / Total Dissolved Solids / Temperature
- pH / Conductivity / Total Dissolved Solids / Salinity / Temperature PCSTestr 35 (Eutech PCSTEST35-01X441506 / Oakton 35425-10)

## **Setting Started:**



best accuracy

Your Testr begins in the measuring mode that was previously used. Just prior to measurement or when switching modes, you will see the setting associated with each parameter i.e.) pH (buffer group selected), Conductivity (Auto), TDS (factor), Salinity (unit of measure).

Your Testr allows customization of various settings. To access the setup mode:

- (Parameter) will appear. With the Testr off, keep the pressed down while you press and release (Setup) will appear, then as you release
  - Press to choose PR-R (Parameter Setup) or 595k (System Setup)
- Press to enter the selected setup menu.

'n

v,

When you are finished making your desired changes, press both 📆 and 🥰 at the same \*IMPORTANT\* It is necessary to save your Parameter and System Men you are finished making your desired chatten.

With the primary display "SA" and secondar chances. The instrument will resume measurem chances. The instrument will resume measurem

to save the With the primary display "SA" and secondary display "YES", press time and keep them pressed until you see "SA" (Save) on the display.

changes. The instrument will resume measurement mode with new setting(s).

Note: If auto-shut off is used, changes will be automatically saved 8.5 minutes after the last

**Parameter Setup:** Select Limit to make changes relating to the parameters – pH, Conductivity TDS, Salinity. Note: only the PCS Testr will have all of these options. See below for menus available from each parameter. To Navigate the PR-R to make changes relating to Parameter Setup: Select menus:

- Press ( to select or confirm the displayed option.
- Press or to scroll thru options or change values.

### pH Options:

- USA or NIST Buffer Group for calibration buffer option.
- 5-pt calibration (all points) or 3-pt calibration (middle three points only).

## Salinity (SALt) Option (PCS Testr only)

Choose PPt (parts per thousand) or Per (percentage %) as unit of measure.

## Total Dissolved Solids (tDS) Option (PT and PCS Tester only)

FACt factor the instrument uses to convert from conductivity to TDS value. Adjustable from 0.40 to 1.00 (default factor is 0.71).

## Conductivity Options (PC and PCS Tester only)

- A.Cal (Automatic Calibration) Choose YES or NO (manual).
- standards that are not listed below, choose "NO" which will disable auto calibration and TIP: The PC Testr 35 and PCS Testr 35 are capable of automatic or manual choose one of (3) conductivity calibration standards depending on the ranges listed automatic calibration is a time saving option. If you intend to calibrate with one or more conductivity calibration. In automatic calibration mode, the meter will automatically below. If you will only use 84 µS, 1413 µS, or 12.88 mS calibration standards, allow you to enter your desired value manually.

On Outcomy range         Automatic calibration value           0.0 – 200.0 μS         84 μS           201 – 2000 μS         1413 μS           201 – 20 00 mS         12 88 mS
0.0 – 200.0 µS 201 – 2000 µS 2.01 – 20.00 mS

 SPC (Single-Point Calibration) Choose YES or NU (multi-point calibration).
 TIP: The PC Testr 35 and PCS Testr 35 are capable of single or multi-point conductivity SPC (Single-Point Calibration) Choose YES or NO (multi-point calibration),

calibration. Use Single-Point Calibration to apply a single calibration value across all ranges. Use Multi-Point Calibration for individual calibration in each range. This will restrict an individual calibration so that it is applied to one range only. When using multi-point calibration, perform a calibration in each range that you expect to use for best results

### System Setup:

Select 595k to make changes relating to the system. See below for available menus. Note: other than changing Temperature units, it is advised to keep the factory default settings for best results. To Navigate the System menus:

Press (1997) to select or confirm the displayed option.

- Press or to scroll thru menu options or change values.
- Unit rSt (Instrument reset)
- PH (pH) or EC (electrical conductivity / TDS / Salinity)
- CAL (calibration reset) or FCt (Reset to factory default settings) Set A.Off (Automatic shut off after 8.5 minutes) Choose YES or NO.
  - Set t.C (Temperature Coefficient) 0.0-10.0% (2.1% is default)
- Set AtC (Auto Temperature Compensation) Choose YES or NO (25°C is used).

  - Set °C °F (select temperature units) Choose °Celsius or °Fahrenheit.

## Temperature Calibration:

accurate thermometer or another Testr. The temperature value is common to all The factory temperature calibration should last for the life of the original sensor since it doesn't normally drift. Temperature calibration is always recommended upon sensor replacement. It may also be desirable to adjust the temperature to match a certified parameters so only one calibration is needed. To perform temperature calibration: Press 🔐 to tum on meter. Place the reference thermometer and your Testr into the same sample. Allow enough time for both to stabilize.

Press 🛗 as needed to select the pH measuring mode. Press 📤 to begin pH

calibration mode.

7

- Press 🍑 for 5 seconds to begin temperature calibration mode. The current temperature will be displayed on top while the factory default temperature is m;
- Press  $\stackrel{\text{dis}}{\longrightarrow}$  or  $\stackrel{\text{dis}}{\longrightarrow}$  to manually adjust to the desired temperature—up to  $\pm~5^\circ$ C or ± 9° F of the factory default value. 4.
- Press 🚾 to confirm and return to the pH measuring mode. 5

### pH Calibration:

For best results, calibrate with certified accurate pH calibration standards (buffers). You may calibrate up to five points with the USA (1.68, 4.01, 7.00, 10.01, 12.45) or best results, calibrate with certified accurate pH calibration standards (buffers). the NIST (1.68, 4.01, 6.86, 9.18, 12.45) buffer group.

- Press to turn meter on and to select pH mode as needed.
- Rinse the sensor with clean water. Immerse the sensor into your pH buffer and - ~
- press 📤. The primary display will show the un-calibrated pH value, while the secondary display should search for and lock on the closest automatic calibration
- Allow the primary display to stabilize, then press we to confirm the calibration value. The primary value will blink briefly before the secondary value automatically scrolls thru the remaining pH buffers available for calibration 'n
- 9 to return Repeat steps 2 & 3 with additional buffers or press 4.

## Conductivity Calibration (Automatic):

Selection of multi-point calibration will allow up to three of the following values, while Single-point calibration will allow only one; choose 84 µS, 1413 µS, or 12.88 mS. For best results, calibrate with certified accurate conductivity calibration standards.

Conductivity Range	Automatic Calibration Value	Available with
0.0 — 200.0 µS	84 hS	PCS only
201 — 2000 µS	1413 µS	PC or PCS
2.01 — 20.00 mS	12.88 mS	PC or PCS

- Rinse the sensor with clean water. Immerse the sensor into your standard and (2) to turn meter on and (2) to select conductivity mode as needed. Press -: ~:
- . The primary display will show the un-calibrated value, while the secondary display display should search for and lock on the closest automatic calibration value. press 🕰
- Allow the primary display to stabilize, then press 🥶 to confirm the calibration value. The primary value will blink briefly before returning to measurement mode. က်
  - Repeat steps 2 & 3 with additional calibrations standards if desired.

## Conductivity, TDS, & Salinity Calibration (Manual):

For best results, calibrate with certified accurate calibration standards. 1 point per

Salinity (1-pt)		1.00 — 10 ppt	
TDS (3-pt)	0.0 — 99.9 ppm*	100 — 999 ppm	1.00 — 10 ppt
Conductivity (3-pt)	0.0 — 200.0 µS*	201 – 2000 µS	2.01 — 20.00 mS

\*Range only available with PCS Testr 35

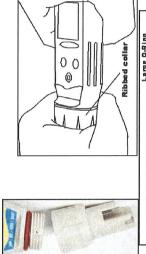
- Press 🐫 to turn meter on and 🐃 to select conductivity, TDS, or salinity <u>.</u>:
- . The primary display will show the un-calibrated value, while the Rinse the sensor with clean water. Immerse the sensor into your standard and secondary display will display the factory default calibration. press 🕰
  - to manually adjust the primary display to your calibration or . standard. Press 'n
- Press 👑 to confirm the new adjusted value. The primary value will blink briefly before returning to measurement mode. 4.
- Repeat steps 2 & 3 with additional calibration standards if desired. ر 0

Fold Function:

Representation of a reading, press when the reading is held. To the "HOLD" indicator will display when the reading is held. To the "Both of the display when the reading is held. To the same live measurement, press

## Sensor Replacement:

Your instrument includes a replaceable sensor (Eutech PCSENSOR - 01X097108 / Oakton 35425-50). If the tip gets damaged or as the sensor wears over time, the entire sensor can easily be replaced. To remove the old sensor, simply twist off the ribbed collar and pull the sensor straight out.



Large O-Ring		Large Tab
		Small Tab
insert Electrode Module	Electrode Module	Small O-Ring
insert El		



sensor to the instrument body. Twist ribbed collar back on to To install the new sensor, line up the tabs and 8 pins of the keep waterproof rating and secure sensor. The 0-rings should create a watertight seal and provide some resistance when twisting.

## Battery Replacement:

Your Testr includes (4) 1.5V alkaline batteries. LR44 or A76 battery types are suitable

Waiting too long to replace the batteries can lead to inaccurate and commonly available. Replace all (4) batteries together.

unscrew to remove the battery cover at the top of the Testr. Pull on the white ribbon to remove the batteries. Note the correct polarity of the instrument before installing. The flat side of the battery is +. Place new batteries on top of the white ribbon so they can be easily removed next time. Hand readings and is the most common cause of problems. Twist and tighten the battery cover to keep waterproof rating.

### Storage:

The sensor does not require special storage. Rinse with clean water after use and cover the sensor with the included cap. Keep at room temperature away from extreme temperatures. The sensor can easily be re-hydrated by soaking if stored dry.

Indicates	>75% battery life remaining	50-75% battery life remaining	25-50% battery life remaining	No bars & blinking = replace batteries	Calibration error, usually attempting to calibrate to a value which is out of range or under range.	Broken or dirty sensor. Clean, rehydrate, and replace if necessary. Could also be due to low battery condition or sample with temperature that has not stabilized.	Measured value is out of range. Most often caused by dry electrode that needs to be re-hydrated / soaked. Sensor may not be completely submersed or is not connected to Testr body properly.	If "Hold" on display, press Hold key to resume live measurement.	The automatic calibration standard is not within expected calibration range. Use fresh standard or an alternate calibration standard.
	>75% ba	50-75%	25-50%	No bars &	Calibration out of ran	Broken or Could also temperati	Measurec that need completel	"Hold"	The autor range. Us
Message				1	Err	Unstable pH reading / Slow response	"Ur" (Under range) or "Or" (Over range)	Meter not responsive	Secondary display continually scrolls

### Warranty

adjustment or replacement is necessary and has not been the result of abuse or and correction will be made without charge. Out of warranty products will be repaired misuse within the time period specified, please return the tester - freight prepaid -The waterproof Testrs are warranted to be free from manufacturing defects for 1 year and the electrode module is warranted for 6 months, unless otherwise stated. If repair, on a charge basis.

## Return of Items:

reason. When applying for authorization, please include information regarding the reason the item(s) are to be returned. Note: We reserve the right to make Authorization must be obtained from your distributor before returning items for any improvements in design, construction and appearance of products without notice. Prices are subject to change without notice.

For more information on our products, please contact us or visit our websites:

#04-16/24, Singapore 139949 Blk 55, Ayer Rajah Crescent, Eutech Instruments Pte Ltd eutech@thermofisher.com Fax: (65) 6773 0836 Tel: (65) 6778 6876 www.eutechinst.com Vernon Hills, IL 60061, USA Fax: (1) 847-247-2984 Tel: (1) 888-462-5866 625 E Bunker Court Oakton Instruments info@4oakton.com www.4oakton.com

### **Attachment D**

Total Residual Chlorine Standard Operating Procedure



### Standard Operating Procedure - Total Residual Chlorine

### 1.0 BREMO VPDES PERMIT TOTAL RESIDUAL CHLORINE ANALYSIS

This standard operating procedure (SOP) is for total residual chlorine (TRC) analysis of discharge water at the Bremo Power Station (Bremo) required for Whole Effluent Toxicity analysis. The HACH Pocket Colorimeter II is used for TRC analysis at 500-series Outfalls.

### 2.0 TOTAL RESIDUAL CHLORINE ANALYSIS

The following procedure is based on the User Manual for the HACH Pocket Colorimeter II, the draft VPDES Permit for Bremo, the VA DEQ check sheet for TRC analysis, and EPA Standard Method 4500-CI G-2000 (SM 21 CI). Analysts performing TRC analysis must be certified in competency for the instrument used on site with an initial demonstration of capability (IDC). The HACH Pocket Colorimeter II and Spec✓™ gel standards are shown below.





### 2.1 Care and Maintenance

- Clean the sample vials included in the kit after each usage with a water and alconox solution followed by a de-ionized water rinse.
- Inspect for scratches or damage on the sample vials prior to each analysis. Replace the kit or vials if the condition is no longer acceptable.
- Inspect the N,N'-diethyl-p-phenylenediamine (DPD) reagent powder pillows stamped expiration date and replace if necessary.
- Inspect the Spec ✓ ™ gel standards expiration date and replace, if necessary.

### 3.0 INITIAL DEMONSTRATION OF CAPABILITY

The initial demonstration of capability (IDC) will be performed once by the analyst/operator to ensure acceptable precision, accuracy, and sensitivity are provided for TRC analysis. The IDC uses the EPA Standard Method 4500-Cl (21st edition) and manufacturers guidance Total Chlorine Analyzer Verification (Attachment III) as a reference and will be performed by each analyst/operator prior to performing a field TRC analysis. The IDC will be documented on the IDC form presented in Attachment IV and performed in the presence of another analyst/operator who has completed the IDC, as a means quality control, using the procedures identified in the Total Chlorine Analyzer Verification Document.

### 4.0 LOW RANGE TRC ANALYSIS FOR HACH POCKET COLORIMETER II

**CAUTION**: The analyst will don disposable nitrile gloves and safety eyewear with side shields for the duration of the analysis due to risk of exposure to DPD reagent and effluent water.





### Standard Operating Procedure - Total Residual Chlorine

- 1. Daily, before use, perform a calibration verification using Spec✓™ gel standards. Record the Specê gel standard expiration date and calibration results on the 24-hour Composite Sampling Log.
- 2. Purge the effluent sample port in accordance with the Sampling and Analysis Plan.
- Document the expiration date for the DPD reagent powder pillows on the Composite Sampling Logs.
- Follow manufacturer procedure for Low Range Total Chlorine EPA DPD Method 8167 (Attachment I).
- 5. Record the result in milligrams per liter (mg/L) on the 24-hour Composite Sampling Log.

Attachment I – Method 8167 Low Range TRC

Attachment II – DEQ TRC Checksheet – Method DPD Colorimetric

Attachment III - Total Chlorine Analyzer Verification

Attachment IV - TRC Initial Demonstration

### Chlorine, Free and Total, Low Range

DOC316.53.01450

### USEPA DPD Method<sup>1</sup> 0.02 to 2.00 mg/L Cl<sub>2</sub> (LR)

Method 8021 (free) 8167 (total)
Powder Pillows or AccuVac® Ampuls

**Scope and application:** For testing free chlorine (hypochlorous acid and hypochlorite ion) in water and treated waters. For testing total chlorine in water, treated waters and wastewater. USEPA accepted for reporting for drinking water analyses.<sup>2</sup> This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.

1 USEPA accepted for reporting wastewater and drinking water analyses.

2 Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-CI G for drinking water.



### Test preparation

### Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis

Always do tests in sample cells or AccuVac<sup>®</sup> Ampuls. Do not put the instrument in the sample or pour the sample into the cell holder.

Make sure that the sample cells are clean and there are no scratches where the light passes through them.

Rinse the sample cell and cap with the sample three times before the sample cell is filled.

Make sure that there are no fingerprints or liquid on the external surface of the sample cells or AccuVac<sup>®</sup> Ampuls. Wipe with a lint-free cloth before measurement.

Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.

After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.

If the test result is over-range, or if the sample temporarily turns yellow after the reagent addition, dilute the sample with a known volume of high quality, chlorine demand-free water and do the test again. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Additional methods are available to measure chlorine without dilution.

The AccuVac Ampul Snapper makes AccuVac Ampul tests easier to do. The AccuVac Ampul Snapper keeps the broken tip of the ampul, prevents exposure to the sample and provides controlled conditions for filling the ampule.

The SwifTest Dispenser for Free Chlorine or Total Chlorine can be used in place of the powder pillow in the test procedures. One dispensation equals one powder pillow for 10-mL samples.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

### Items to collect

### Powder pillows

Description	Quantity
Chlorine, Free: DPD Free Chlorine Reagent Powder Pillows, 10-mL	1
Sample cells, 25-mm (10 mL)	2

Refer to Consumables and replacement items on page 7 for order information.

### AccuVac Ampuls

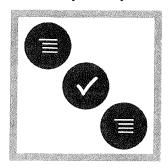
Description	Quantity
Chlorine, Free: DPD Free Chlorine Reagent AccuVac Ampuls	1
Chlorine, Total: DPD Total Chlorine Reagent AccuVac Ampuls	1
Beaker, 50-mL	1
Stopper for 18-mm tubes and AccuVac Ampuls	1
Sample cells, 25-mm (10 mL)	1

Refer to Consumables and replacement items on page 7 for order information.

### Sample collection

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and is unstable in natural waters. Chlorine reacts
  quickly with various inorganic compounds and more slowly with organic compounds.
  Many factors, including reactant concentrations, sunlight, pH, temperature and
  salinity influence the decomposition of chlorine in water.
- Collect samples in clean glass bottles. Do not use plastic containers because these can have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand. Soak the containers in a weak bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse fully with deionized or distilled water. If sample containers are rinsed fully with deionized or distilled water after use, only occasional pretreatment is necessary.
- Make sure to get a representative sample. If the sample is taken from a spigot or faucet, let the water flow for at least 5 minutes. Let the container overflow with the sample several times and then put the cap on the sample container so that there is no headspace (air) above the sample.

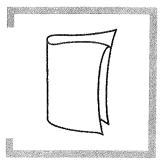
### Powder pillow procedure



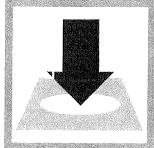
1. Set the instrument to low range (LR). Refer to the instrument documentation.



2. Prepare the blank: Fill a sample cell to the 10-mL mark with sample. Close the sample cell.



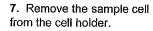
3. Clean the blank sample cell.

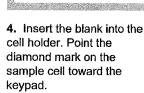


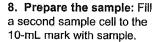


**5.** Install the instrument cap over the cell holder.



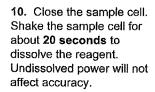




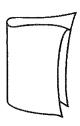




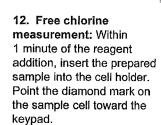
9. Add one 10-mL DPD Free Chlorine Reagent Powder Pillow or one 10-mL DPD Total Chlorine Reagent Powder Pillow to the second sample cell.



A pink color will show if chlorine is in the sample.



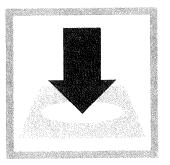
**11.** Clean the prepared sample cell.



Go to step 15.



**13.** Set and start a timer for 3 minutes. A 3-minute reaction time starts.



14. Total chlorine measurement: After 3 minutes and within 6 minutes of the reagent addition, insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.

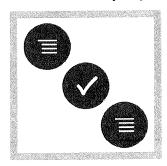


**15.** Install the instrument cap over the cell holder.



**16.** Push READ. Results show in mg/L Cl<sub>2</sub>.

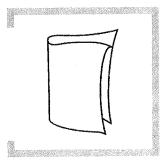
### AccuVac® Ampul procedure



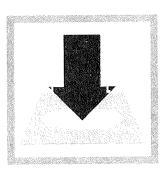
1. Set the instrument to low range (LR). Refer to the instrument documentation.



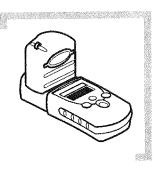
2. Prepare the blank: Fill a sample cell to the 10-mL mark with sample. Close the sample cell.



3. Clean the blank sample cell.



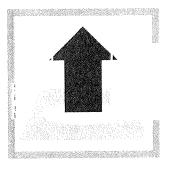
4. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



Install the instrument cap over the cell holder.



**6.** Push **ZERO**. The display shows "0.00".



7. Remove the sample cell from the cell holder.



8. Prepare the sample:
Collect at least 40 mL of sample in a 50-mL beaker.
Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.









03:00

**9.** Quickly invert the AccuVac Ampul several times to mix.

**10.** Clean the AccuVac Ampul.

11. Free chlorine measurement: Within 1 minute of the reagent addition, insert the prepared sample AccuVac Ampul into the cell holder.
Go to step 15.

**12.** Set and start a timer for 3 minutes. A 3-minute reaction time starts.



**13.** When the timer expires, clean the prepared sample cell.

14. Total chlorine measurement: Within 6 minutes of the reagent addition, insert the prepared sample AccuVac Ampul into the cell holder.

**15**. Install the instrument cap over the cell holder.

**16.** Push READ. Results show in mg/L Cl<sub>2</sub>.

### Interferences

Interfering substance	Interference level
Acidity	More than 150 mg/L CaCO <sub>3</sub> . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sodium Hydroxide. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Alkalinity	More than 250 mg/L CaCO <sub>3</sub> . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sulfuric Acid. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Bromine, Br <sub>2</sub>	Positive interference at all levels
Chlorine Dioxide, CIO <sub>2</sub>	Positive interference at all levels
Inorganic chloramines	Positive interference at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as CaCO <sub>3</sub>

Interfering substance	Interference level
Manganese, Oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, Oxidized (Cr <sup>6+</sup> )	<ol> <li>Adjust the sample pH to 6–7.</li> <li>Add 3 drops of Potassium lodide (30-g/L) to 10 mL of sample.</li> <li>Mix and wait 1 minute.</li> <li>Add 3 drops of Sodium Arsenite (5-g/L) and mix.</li> <li>Use the test procedure to measure the concentration of the treated sample.</li> <li>Subtract this result from the result without the treatment to obtain the correct chlorine concentration.</li> </ol>
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Positive interference at all levels
Peroxides	May interfere
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. Adjust to pH 6–7 with acid (Sulfuric Acid, 1 N) or base (Sodium Hydroxide, 1 N). Correct the test result for the dilution caused by the volume additions.

### Pollution prevention and waste management

If sodium arsenite was added to the sample for manganese or chromium interferences, the reacted samples will contain arsenic and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations. must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

### Accuracy check

### Standard additions method

Use the standard additions method to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Chlorine Standard Solution, 2-mL PourRite® Ampule, 25–30 mg/L (use mg/L on label)
- Ampule breaker
- Pipet, TenSette<sup>®</sup>, 0.1–1.0 mL and tips
- Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

**Note:** For AccuVac<sup>®</sup> Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the standard solution to three 50-mL portions of fresh sample.

- 2. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
- 3. Compare the expected result to the actual result. The expected increase in the chlorine concentration is the Cl<sub>2</sub> mg/L concentration from the label of the standard solution multiplied by 0.1 mL for every 10 mL of standard solution added.

### Standard solution method

If the Standard Calibration Adjust feature is used to adjust the calibration curve of the Pocket Colorimeter II, the concentration of the chlorine standard must be between 0.50 and 1.50 mg/L chlorine for the LR procedure.

### Verification of on-line analyzers

This procedure can be used to meet the requirements of USEPA Method 334.0 - Determination of Residual Chlorine in Drinking Water Using an On-line Chlorine Analyzer.

ANALYST:	VPDES NO.	

### Parameter: Total Residual Chlorine (TRC) Method: DPD Colorimetric (HACH Pocket and Pocket II Colorimeter) 11/2014

METH	OD OF ANALYSIS: Instrument:		
	HACH Manufacturer's Instructions (Method 8167) plus an edition of Standard Methods		
	21st Edition of Standard Methods 4500-Cl G-2000 (SM 21 Cl)		
	22 <sup>nd</sup> Edition of <i>Standard Methods</i> 4500-Cl G-2011 (SM 22 Cl)		
		Υ	N
1)	Is a certificate of operator competence or initial demonstration of capability available for <u>each</u> <u>analyst/operator</u> performing this analysis? <b>NOTE:</b> Analyze 4 samples of known TRC. Must use a lot number or source that is different from that used to prepare calibration standards. May not use Spec√ <sup>™</sup> . Acceptance range is 70-130% recovery <u>and</u> 20% Relative Standard Deviation (RSD) <u>or</u> within PT specified acceptance range <u>and</u> 20% RSD. [SM 1020 B.1]		
2)	Is calibration curve verification checked daily using a high and a low standard? <b>NOTE:</b> May use manufacturer's installed calibration and commercially available chlorine standards, or Spec√ ™, for daily calibration verifications. [SM 21 1020]		
3)	<u>IF</u> a replicate sample is analyzed is there a written procedure for which result will be reported on DMR (Sample or Replicate) and is this procedure being followed? [DEQ – based on EPA Good Laboratory Practices Standards]		
4)	Is a Laboratory Control Sample (LCS) tested at least annually and are results within acceptance criteria? [SM 21 B. 2. or SM 22 1020 B 3.] <b>NOTE:</b> LCS should be a purchased Proficiency Test (PT) sample or if a known standard different from the calibration standards is used. Use the PT acceptance criteria when given or use 70-130% recovery <u>and</u> 20% Relative Standard Deviation (RSD) as the acceptance criteria.		
5)	Are the DPD Powder Pillows stored in a cool, dry place? [Mfr.]		
6)	Are the pillows within the manufacturer's expiration date? [Mfr.]		
7	Are pillows appropriate for the sample size being analyzed and for <u>Total</u> Residual Chlorine		
8)	Has buffering capability of DPD pillows been checked annually? (Pillows should adjust sample pH to between 6 and 7) [Mfr.]		
9)	When pH adjustment is required, is H <sub>2</sub> SO <sub>4</sub> or NaOH used? [Hach 11.3.1]		
10)	Are cells clean and in good condition? [Mfr]		
11)	Is the Hach colorimeter program set to measure "TRC, mg/L"? [Mfr.]		
12)	Is the low range (0.01 mg/L resolution) used for samples containing residuals from 0.1 mg/L - 2.00 mg/L? [Mfr.]		
13)	Is the 10-mL cell (2.5-cm diameter) used for samples from 0-2.00 mg/L? [Mfr.]		
14)	Are samples analyzed within 15 minutes of collection? [40 CFR Part 136]		
15)	Is meter zeroed correctly using sample for the blank analysis? [Mfr. and SM 21 1020 B.4. or SM 22 1020 B.5.]]		
16)	Is the instrument light screen placed correctly on the meter body when the meter is zeroed and when the sample is analyzed? [Mfr.]		
17)	Is the DPD Total Chlorine Powder Pillow mixed into the sample? [Hach 11.1]		

18)	Is the analysis made at least three minutes but not more than six minutes after Powder Pillow addition? [Hach 11.2]	
19)	If read-out exceeds "2.19 mg/L", is the original sample diluted correctly, and then reanalyzed within 15 minutes of the original collection time? [Hach 1.2 & 2.0]	

PROBLEMS:

### Free and total chlorine analyzer verification

DOC316.53.01302

0.05 to 4.00 mg/L as Cl<sub>2</sub>

This document contains test procedures to support USEPA Method 334.0, *Determination of Residual Chlorine in Drinking Water using an On-line Chlorine Analyzer*. It is primarily intended to be used by drinking water facilities for compliance with daily monitoring requirements. The appropriate section of the USEPA method is referenced in each test procedure.

### **USEPA Method 334.0 summary:**

An online chlorine analyzer is used to continuously monitor the chlorine concentration at a drinking water sample point. The instrument is calibrated using aqueous standards or the results from paired grab samples that are collected at the same sample point and time. The grab samples are analyzed for chlorine (free or total) using a method that is approved for drinking water compliance monitoring. The online analyzer accuracy is periodically verified/adjusted based on results from grab sample analyses.

### Prerequisites:

- Read the procedures to make sure that all labware is properly treated, the correct reagent quality is available and the procedures and calculations are clearly understood.
- Read the details and precautions in the procedures to make sure that the precision and accuracy requirements of Method 334.0 are achieved.
- Make a decision about whether free chlorine or total chlorine will be measured.
- Make sure that the specified reagents are available.
- Read the specific instrument procedures for chlorine determination (Method 10245, DPD Free Chlorine, or Method 10250, DPD Total Chlorine) for additional test details.
- Use dedicated labware and reagents to reduce preparation and analysis time and to prevent unintentional analysis errors.
- Find out if additional test requirements have been added by the primacy agency.

### Test procedures:

- A. Verification of the chlorine calibration used for grab samples on page 1
- B. Verification of secondary DPD Spec Check chlorine standards on page 5
- C. Initial demonstration of capability (IDC) for field samplers on page 6
- D. Initial calibration/verification of online chlorine analyzers on page 8
- E. Initial demonstration of capability (IDC) for online chlorine analyzers on page 9
- F. Routine verification of the calibration used for grab samples on page 11
- G. Routine verification of the calibration for online chlorine analyzers on page 12

### Start-up procedures

### A. Verification of the chlorine calibration used for grab samples

EPA Method 334.0 reference: Start-up procedures section—grab sample method, paragraph 10.1.1

**Scope and application:** A requirement of USEPA Method 334.0, Section 10.1.1, is to verify the internal, factory-set calibration curve for grab-sample instruments that are used to measure the chlorine concentration of water samples. Three chlorine standard solutions that span the expected concentration range and one method blank must be used. The lowest concentration standard must be at or below 0.2 mg/L, or at or below the minimum chlorine residual requirement set by the primacy agency. Hach DPD Methods 10245 and 10250 for free or total chlorine, 0–4 mg/L  $\rm Cl_2$ , use internal factory-set calibration curves. These curves are verified using the following procedure.

### Before starting the test:

Calculate and record the concentration and dilution ratios for the required chlorine standard solutions. Refer to How to calculate the concentration of chlorine standard solutions on page 3.

Use only chlorine demand-free glassware and organic-free or chlorine demand-free dilution water.

Analyze chlorine standards immediately after preparation.

Follow the analysis procedure for Method 10245 for free chlorine or Method 10250 for total chlorine.

### Test procedure

Preparation and analysis of the chlorine verification standards

- 1. Add approximately 25 mL of organic-free water into a clean 50-mL or 100-mL volumetric flask.
- 2. Open a chlorine standard solution ampule, 50-75 mg/L Cl<sub>2</sub>.
- 3. Use a pipettor to add the calculated volume of the standard solution into the flask. Refer to How to calculate the concentration of chlorine standard solutions on page 3.
- **4.** Dilute to the mark with organic-free water. Stopper and invert several times to mix.
- 5. Repeat as needed for preparation of additional standard concentrations.
- **6.** Select the program number or channel for Method 10245 or Method 10250 DPD Free or Total Chlorine on the grab-sample instrument.
- 7. Fill a sample cell with organic-free water to set the instrument zero. Insert the sample cell into the instrument and push ZERO. The display shows 0.00 mg/l Cl<sub>2</sub>.
- **8.** Add 1 DPD Free Chlorine or 1 DPD Total Chlorine Reagent Powder Pillow for a 25-mL sample to 10 mL of organic-free water (method blank). Cap and invert to dissolve the reagent.

**Note:** DPD Total Chlorine Reagent may be used to verify the calibration curve for both free and total chlorine determinations.

- **9.** Insert the prepared method blank into the instrument. Read and record the value at the time that is specified in the method test procedure.
- **10.** Repeat step 8 for each of the prepared standards.
- **11.** Insert the prepared standards into the instrument. Read and record the result at the time that is specified in the method test procedure.
- 12. Compare the measured values of the prepared standards to the calculated values.

If the measured values are within  $\pm 15\%$  of the calculated values for the prepared chlorine standard solutions, the calibration curve is successfully verified. The measured value for the method blank must be equal to or less than  $^{1}/_{3}$  the concentration of the lowest standard used. If the measured values for the chlorine standards or method blank do not meet the acceptance criteria, refer to Troubleshooting when grab sample verification criteria is not met on page 3.

### Example:

Three standard solutions were prepared. The calculated and measured chlorine concentrations are shown in Table 1. The measured concentrations are within the ±15% allowable range. The internal factory calibration curve is successfully verified.

Table 1 Comparison of calculated and measured standard values (in mg/L Cl<sub>2</sub>)

Standard	Calculated concentration	Allowable range (±15%)	Measured concentration	Status
Blank		0.00-0.061	0.01 mg/L	Pass
Std 1	0.20 mg/L	0.17-0.23	0.21 mg/L	Pass
Std 2	1.60 mg/L	1.36–1.84	1.62 mg/L	Pass
Std 3	3.50 mg/L	2.98-4.03	3.49 mg/L	Pass

<sup>1 1/3</sup> or less of the Standard 1 value

### Troubleshooting when grab sample verification criteria is not met

If the verification criteria for grab samples is not met:

- Repeat the test procedure to confirm the results.
- Make sure that the glassware and water that is used is free of chlorine demand.
- Refer to the user manual for the grab sample instrument and complete the Calibration Adjust
  procedure. Use a chlorine standard concentration that is near the mid range of the test
  method. After the Calibration Adjust procedure is complete, repeat the test procedure for
  verification of the grab sample method.

### How to calculate the concentration of chlorine standard solutions

Chlorine standard solutions are prepared by dilution of a concentrated (50 to 75 mg/L Cl<sub>2</sub>) chlorine standard contained in ampules. The actual concentration changes for each lot of standard and is printed on the package label.

1. Calculate the volume of standard solution to be pipetted from the ampule:

<u>Desired final chlorine concentration × Volumetric flask volume</u>
Ampule standard concentration (from label)

Estimated pipetted volume

- 2. Round the estimated volume to the nearest 0.05 mL (most pipettors read to 0.05 mL only).
- 3. Calculate the actual concentration of the diluted standard solution:

Actual pipet volume × Ampule standard concentration (from label) = Diluted standard concentration Volumetric flask volume

### Example:

Desired chlorine concentration: 1 mg/L Cl<sub>2</sub>

Ampule standard concentration from label: 65.3 mg/L Cl<sub>2</sub>

Volumetric flask volume: 50 mL

Estimated volume of ampule standard solution to be used:

$$\frac{1 \text{ mg/L} \times 50 \text{ mL}}{65.3 \text{ mg/L}} = 0.766 \text{ mL}$$

Concentration of diluted standard using actual pipette volume:

$$\frac{0.75 \text{ mL} \times 65.3 \text{ mg/L}}{50 \text{ ml}} = 0.98 \text{ mg/L}$$

Use a pipettor to add 0.75 mL of a 65.3 mg/L chlorine standard solution into a 50-mL volumetric flask. Dilute to the mark with chlorine demand-free water to make a 0.98 mg/L chlorine standard.

Additional examples of chlorine standard solutions are shown in Table 2.

Table 2 Examples of volumes used to prepare chlorine standard solutions

Desired concentration	Ampule standard concentration	Volumetric flask volume	Calculated pipette volume	Actual pipette volume	Final concentration
0.10 mg/L	65.3 mg/L	100 mL	0.153 mL	0.20 mL	0.13 mg/L
0.20 mg/L	65.3 mg/L	100 mL	0.306 mL	0.30 mL	0.20 mg/L
1.00 mg/L	65.3 mg/L	50 mL	0.766 mL	0.75 mL	0.98 mg/L
2.00 mg/L	65.3 mg/L	50 mL	1.531 mL	1.55 mL	2.02 mg/L
4.00 mg/L	65.3 mg/L	50 mL	3.062 mL	3.05 mL	3.98 mg/L

### Treatment of glassware for chlorine demand

Pretreat glass sample containers to remove any chlorine demand by soaking the container in a dilute bleach solution (1 mL commercial bleach in 1 L of deionized water) for at least 1 to 3 hours. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

### Sample cells

Do not use the same sample cells for free and total chlorine testing. A trace of iodide from the total chlorine reagent can contaminate the free chlorine test and cause a monochloramine interference. It is best to use separate, dedicated sample cells for free and total chlorine measurements.

### Chlorine demand-free water

Hach Company organic-free water is acceptable for the preparation of chlorine standards and eliminates the need to prepare chlorine-demand free water. Procedures for the preparation of chlorine demand-free water are included in ASTM-D 1253-06 and Standard Method 4500-CI C.

### Replacement parts and reagents

### Reagents

Description	Unit	Catalog number
DPD Free Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1407099
or		
DPD Total Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1406499
Chlorine Standard Solution, 2-mL PourRite® Ampule, 50–75 mg/L	20/pkg	1426820
or		
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	1426810
DPD Chlorine-MR Spec Check Secondary Standards Kit	each	2980500
Organic-free water	500 mL	2641549

### **Apparatus**

Description	Unit	Catalog number
Ampule Breaker Kit, 10-mL Voluette Ampules	each	2196800
Ampule Breaker, 2-mL PourRite Ampules	each	2484600
Flask, volumetric, 50 mL	each	1457441
Flask, volumetric, 100 mL	each	1457442
Gloves, chemical resistant, size 10 <sup>1</sup>	each	2410105
Goggles, safety, standard	each	2927902
Notebook, field	each	2091800
Pipet, variable volume, 0.2–1.0 mL	each	BBP078

### Apparatus (continued)

Description	Unit	Catalog number
Pipet Tips, for BBP078 pipet	100/pkg	BBP079
or		
Pipet, variable volume, 1.0–5.0 mL	each	BBP065
Pipet Tips, for BBP065 pipet	75/pkg	BBP068
Water, deionized	4 L	27256
Wipes, disposable, 28 x 37 cm	188/pkg	2932800

Additional sizes available

### B. Verification of secondary DPD Spec Check chlorine standards

EPA Method 334.0 reference: Start-up procedures section—grab sample method, paragraph 10.1.1.3

Scope and application: USEPA Method 334.0 allows the use of secondary standards to support the grab sample method. The standards must be verified before use by measurement in an instrument with a calibration curve that was recently verified with grab samples. The Hach DPD Chlorine-MR Spec Check Secondary Standards are designed to support the grab sample testing requirements in Method 334.0. The secondary standards must be verified for each grab sample instrument that will be used. Specific chlorine values are assigned to each Chlorine Spec Check Secondary Standard at the time of verification on a verified chlorine grab-sample calibration curve. During routine use, the assigned values for the Spec Check standards must be within ±10% of their assigned value when compared to the value obtained on the grab sample instrument.

### Before starting the test:

Use the DPD Chlorine-MR Spec Check Kit with Hach Method 10245 for DPD Free Chlorine and Method 10250 for DPD Total Chlorine.

Review the Spec Check kit instructions printed on the kit label and on the certificate of analysis.

Use the Spec Check blank vial included with the kit.

Record the instrument identification and program number directly on the Record of Performance Verification included with the kit.

One Record of Performance Verification is required for each grab sample instrument.

It is best to obtain a set of Spec Check standards for each grab sample instrument, so that they will be available for calibration verification at all times.

Secondary standards must be also be verified each time a grab-sample instrument calibration curve is changed.

Secondary standards must not be used beyond the expiration date on the kit label.

### Test procedure

- 1. Select the grab-sample instrument program for 0–4 mg/L chlorine, DPD Method 10245 or Method 10250, on the instrument.
- 2. Wipe each Spec Check vial with a clean tissue.
- 3. Insert the Spec Check Blank into the instrument and push ZERO.
- 4. Insert STD 1 into the instrument and push READ to show the chlorine value.
- 5. Write the chlorine value on the Record of Performance Verification label.

- 6. Repeat steps 4 and 5 with STD 2 and STD 3.
- 7. Attach the completed Record of Performance Verification label to the instrument.

When the chlorine values are assigned from the verified grab sample instrument, the verification of the secondary standards is complete. Subsequent measurements on the same instrument should be within 10% of the assigned value.

### Replacement parts and reagents

### Reagents and apparatus

Description	Unit	Catalog number
DPD Chlorine-MR Spec Check Secondary Standards Kit	each	2980500
Wipes, disposable, 28 x 37 cm	188/pkg	2932800

### C. Initial demonstration of capability (IDC) for field samplers

EPA Method 334.0 reference: Start-up procedures section—grab sample method, paragraph 10.1.2

**Scope and application:** Each field sampler or person responsible for verification of online chlorine analyzers must first complete an initial demonstration of capability (IDC) before the grab sample method can be used to verify the accuracy of online chlorine analyzers. The IDC requires both an accuracy and precision calculation. Five replicates of a method blank and five replicates of a mid-range chlorine standard are run. The average concentration of the five replicates of the mid-range chlorine standard must be within ±15% of the expected chlorine concentration. The average value of the five method blank values must be equal to or less than <sup>1</sup>/<sub>3</sub> the value of the lowest chlorine concentration used to initially verify the grab sample instrument. The precision or scatter in the concentration values of the five chlorine standard replicates must have a relative standard deviation (RSD) equal to or less than 15% of the average value.

### Before starting the test:

Read the procedure to understand the testing requirements and mathematical calculations required to complete the Initial Demonstration of Capability.

A spreadsheet application can be used to calculate the statistics.

Laboratory personnel can prepare the independent reference samples for analyses by the field samplers.

Prepare glassware that is free of chlorine demand before preparation of the chlorine standard.

Analyze the five replicate chlorine standards immediately.

Use separate sample cells for free and total chlorine analysis to prevent contamination.

Maintain a record of the IDC results for each field sampler.

### Test procedure—method blank accuracy

- 1. Collect the necessary chlorine demand-free glassware, pipets, organic-free water and the Chlorine Standard Ampule Solution.
- 2. Power on the instrument and select the program number or channel for the 0–4 mg/L DPD chlorine method.
- 3. Rinse a clean sample cell two times with organic-free water and then fill to the 10-mL mark.
- 4. Insert the sample cell into the instrument and push ZERO. The display shows 0.00 mg/L Cl<sub>2</sub>.
- **5.** Remove the sample cell and add the contents of 1 DPD Free Chlorine or 1 DPD Total Chlorine Powder Pillow. Cap and shake gently to mix.

- **6.** Insert the prepared method blank into the instrument. Read the value at the time that is specified in the instrument test procedure.
- 7. Record the value and dispose of the solution.
- 8. Repeat steps 3–7 four additional times with the same sample cell to obtain the five replicate method blank values.
- 9. Calculate the accuracy and precision. Refer to Calculate the accuracy and precision.

### Test procedure—chlorine standard accuracy

- Calculate the volume of 50–75 mg/L chlorine standard that is necessary to make an approximate 2 mg/L Cl<sub>2</sub> standard solution. Approximately 100 mL of the diluted chlorine standard solution is required to complete the precision study. Refer to How to calculate the concentration of chlorine standard solutions on page 3.
- 2. Add approximately 25 mL of organic-free water to a clean 100-mL volumetric flask.
- 3. Open a Chlorine Standard Solution Ampule, 50–75 mg/L Cl<sub>2</sub>.
- **4.** Pipette the volume of the chlorine standard solution that was calculated in step 1 into the flask.
- 5. Fill the flask to the mark with organic-free water, stopper and gently invert several times to mix.
- **6.** Immediately rinse a clean sample cell two times with a small portion of the diluted chlorine standard. Fill the sample cell to the 10-mL mark with the diluted standard.
- 7. Insert the sample cell into the instrument and push ZERO. The display shows 0.00 mg/L Cl<sub>2</sub>.
- **8.** Remove the sample cell and add the contents of 1 DPD Free Chlorine or 1 DPD Total Chlorine Powder Pillow for 25 mL samples. Cap and shake gently to mix.
- **9.** Insert the prepared standard into the instrument. Read the result at the time that is specified in the instrument test procedure.
- **10.** Record the value and dispose of the solution.
- **11.** Repeat steps 6–10 four additional times with the same sample cell to obtain the 5 replicate mid-range chlorine standard values.
- 12. Calculate the accuracy and precision. Refer to Calculate the accuracy and precision.

### Calculate the accuracy and precision

### Accuracy:

Average (X) = 
$$\frac{\text{Sum of 5 replicate values}}{5}$$

Average (Method Blank) =  $\frac{\text{Sum of 5 replicate method blank values}}{5}$ 

### Precision:

- 1. Use a spreadsheet application or a calculator function to determine the standard deviation (S) of the five replicate chlorine standard values.
- 2. Calculate the Relative Standard Deviation (RSD):

$$RSD = \frac{S}{X} \times 100\%$$

where:

S = the standard deviation for the 5 replicate standards

X = the average value of the 5 replicate chlorine standards

### Replacement parts and reagents

### Reagents

Description	Unit	Catalog number
DPD Free Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1407099
or		
DPD Total Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1406499
Chlorine Standard Solution, 2-mL PourRite® Ampule, 50–75 mg/L	20/pkg	1426820
or		
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	1426810
DPD Chlorine-MR Spec Check Secondary Standards Kit	each	2980500
Organic-free water	500 mL	2641549

### **Apparatus**

Description	Unit	Catalog number
Ampule Breaker Kit, 10-mL Voluette Ampules	each	2196800
Ampule Breaker, 2-mL PourRite Ampules	each	2484600
Flask, volumetric, 50 mL	each	1457441
Flask, volumetric, 100 mL	each	1457442
Gloves, chemical resistant, size 10 <sup>1</sup>	each	2410105
Goggles, safety, standard	each	2927902
Notebook, field	each	2091800
Pipet, variable volume, 0.2–1.0 mL	each	BBP078
Pipet Tips, for BBP078 pipet	100/pkg	BBP079
or		
Pipet, variable volume, 1.0–5.0 mL	each	BBP065
Pipet Tips, for BBP065 pipet	75/pkg	BBP068
Water, deionized	4 L	27256
Wipes, disposable, 28 x 37 cm	188/pkg	2932800

<sup>&</sup>lt;sup>1</sup> Additional sizes available

### D. Initial calibration/verification of online chlorine analyzers

EPA Method 334.0 reference: Start-up procedures section—online analyzer, paragraph 10.2.1

**Scope and application:** For the initial calibration or verification of the calibration of a newly installed online chlorine analyzer. After the installed analyzer shows stable values, grab samples are taken and analyzed for chlorine with a DPD chlorine method that has been recently verified. The values obtained with the grab sample method are used to verify the analyzer calibration curve.

### Before starting the test:

Identify a collection point for the grab sample that is as close as feasible to the location where the sample enters the online chlorine analyzer.

Collect the necessary instrument, reagents, labware and procedures for the grab sample test method.

Find the user instructions for the online analyzer that includes adjustment of the calibration curve.

### Test procedure

- 1. Measure each of the DPD Chlorine-MR Spec Check Secondary Standards in the colorimeter or spectrophotometer that is used to measure grab samples (section B on page 5). Acceptable readings verify that the instrument performance is good and has not changed after transport to the analyzer site.
- 2. Rinse a glass beaker or flask several times with the chlorine sample and then collect the sample in the beaker or flask.
- 3. Use the 0–4 mg/L DPD method (10245 or 10250) to measure the chlorine concentration of the sample with the grab-sample instrument. Use the same analysis techniques and precautions that were used for the Initial Demonstration of Capability (IDC) grab sample procedure (refer to procedure C on page 6).
- **4.** Record the chlorine concentration of the grab sample. Compare this value to the value from the online analyzer.
- 5. If the measured value of the grab sample is different from the measured value of the online analyzer by more than 15%, adjust the calibration curve of the analyzer to match the grab sample chlorine value.

**Note:** During routine operation of the analyzer, the readings must be within  $\pm 0.1$  mg/L or  $\pm 15\%$  of the grab sample value. Use this criterion as a guide for deciding when the analyzer is properly adjusted.

**6.** If the calibration of the analyzer was adjusted, repeat steps 2–5 until the reading from the chlorine analyzer agrees with the grab sample measurements.

### Replacement parts and reagents

### Reagents

Description	Unit	Catalog number
DPD Free Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1407099
or		
DPD Total Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1406499
DPD Chlorine-MR Spec Check Secondary Standards Kit	each	2980500
Water, deionized, 4 L	27256	Water, deionized

### **Apparatus**

Description	Unit	Catalog number
Beaker, 100 mL, glass	each	50042H
Gloves, chemical resistant, size 10 <sup>1</sup>	each	2410105
Goggles, safety, standard	each	2927902
Notebook, field	each	2091800
Wipes, disposable, 28 x 37 cm	188/pkg	2932800

Additional sizes available

### E. Initial demonstration of capability (IDC) for online chlorine analyzers

EPA Method 334.0 reference: Start-up procedures section—online analyzer, paragraph 10.2.2

**Scope and application:** The initial demonstration of capability (IDC) can only be started after the calibration verification (section D on page 8) is complete. The IDC takes a minimum of 14 days. The full IDC must be completed before the analyzer is used for compliance monitoring measurements. The data collected during the IDC must be recorded and maintained.

### Before starting the test:

The IDC is not required for previously installed chlorine analyzers if historical data shows agreement with grab sample analyses. Agreement is defined as being within ±0.1 mg/L or ±15% (whichever is greater) of the grab sample data. Historical data must show agreement for 14 continuous days without analyzer maintenance or calibration.

For installations with multiple analyzers, the first analyzer must successfully complete the 14-day IDC. Agreement criterion for multiple analyzers is defined as being within ±0.1 mg/L or ±15% (whichever is greater) of the grab sample data.

The IDC for the installation of multiple analyzers can be reduced to seven consecutive days or seven business days when the same model of analyzer is installed at each location. The water characteristics and treatment process must be the same at each location.

Make sure that the calibration for grab sample measurements is verified before the IDC process is started.

Make sure that the IDC requirements for grab sample measurements with the field sampler is completed before the IDC process is started.

### Test procedure

- 1. Analyze two grab samples for chlorine using the program, instrument and procedure of a recently verified measurement system. The samples should be collected at concentrations that represent the daily chlorine high and low values as indicated by the analyzer.
- 2. The data for the two grab samples must be within the  $\pm 0.1$  mg/L or  $\pm 15\%$  acceptance criterion of the analyzer values.
- 3. Repeat steps 1–2 for 14 consecutive days or 14 consecutive business days.
- **4.** When 14 consecutive days of data pairs meet the acceptance criterion, the analyzer can be put into service for compliance monitoring.

**Note:** When operation is stopped for analyzer maintenance, the accuracy of the analyzer must be verified with a grab sample. The accuracy must be verified again after one day of operation.

### Replacement parts and reagents

### Reagents

Description	Unit	Catalog number
DPD Free Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1407099
or		
DPD Total Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1406499
DPD Chlorine-MR Spec Check Secondary Standards Kit	each	2980500
Water, deionized, 4 L	27256	Water, deionized

### **Apparatus**

Description	Unit	Catalog number
Beaker, 100 mL, glass	each	50042H
Gloves, chemical resistant, size 10 <sup>1</sup>	each	2410105
Goggles, safety, standard	each	2927902
Notebook, field	each	2091800
Wipes, disposable, 28 x 37 cm	188/pkg	2932800

<sup>&</sup>lt;sup>1</sup> Additional sizes available

### **Routine Monitoring Procedures**

### F. Routine verification of the calibration used for grab samples

EPA Method 334.0 reference: Routine procedures section—grab sample method, paragraph 11.1

**Scope and application:** This procedure verifies the grab sample method that is used to monitor the online chlorine analyzer during normal operation. The grab sample method must be verified at a minimum of once quarterly or whenever a grab sample is used to adjust the calibration of the online analyzer.

### Before starting the test:

Collect the necessary chlorine demand-free glassware, pipets, organic-free water and the Chlorine Standard Ampule Solution.

An aqueous chlorine check standard is prepared at a concentration near the expected chlorine concentration of the water samples.

The results of the check standard must be within ±15% of the calculated value. If the results are different by more than 15%, the problem must be corrected before the grab sample method can be used to verify online measurements.

DPD Free Chlorine Reagent Powder Pillows or DPD Total Chlorine Reagent Powder Pillows for a 25-mL sample size are used.

The results of the check standard measurement must be recorded and maintained according to the requirements of the primacy agency.

Secondary DPD Chlorine-MR Spec Check Standards can be used to make sure that the performance of the grab sample instrument has not changed.

### Test procedure

- 1. Calculate the volume of a 50–75 mg/L chlorine standard that is necessary to make a chlorine standard solution that is near the typical concentration of the water sample. Refer to How to calculate the concentration of chlorine standard solutions on page 3.
- 2. Power on the instrument that is used for grab sample measurements and select the program number or channel for the 0–4 mg/L chlorine method.
- 3. Add approximately 25 mL of organic-free water to a clean 50-mL or 100-mL volumetric flask.
- 4. Open a Chlorine Standard Solution Ampule, 50–75 mg/L Cl<sub>2</sub>.
- 5. Pipette the volume of the chlorine standard solution that was calculated in step 1 into the flask.
- 6. Fill the flask to the mark with organic-free water, stopper and gently invert several times to mix.
- 7. Immediately rinse a clean sample cell 2 times with a portion of the diluted chlorine standard. Fill the sample cell to the 10-mL line with the diluted standard.
- 8. Insert the sample cell into the instrument and push ZERO. The display shows 0.00 mg/L Cl<sub>2</sub>.
- 9. Remove the sample cell and add the contents of 1 DPD Free Chlorine or 1 DPD Total Chlorine Powder Pillow. Cap and shake gently to mix.
- **10.** Insert the prepared standard into the instrument. Read the result at the time that is specified in the instrument test procedure.
- 11. Record the value and dispose of the solution.
- **12.** Compare the measured value to the calculated value. If the measured value is within ±15% of the calculated value, the grab sample method can be used to verify online analyzer accuracy.

### Replacement parts and reagents

### Reagents

Description	Unit	Catalog number
DPD Free Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1407099
or		
DPD Total Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1406499
Chlorine Standard Solution, 2-mL PourRite® Ampule, 50–75 mg/L	20/pkg	1426820
or		
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	1426810
DPD Chlorine-MR Spec Check Secondary Standards Kit	each	2980500
Organic-free water	500 mL	2641549
Water, deionized	4 L	27256

### **Apparatus**

Description	Unit	Catalog number
Ampule Breaker Kit, 10-mL Voluette Ampules	each	2196800
Ampule Breaker, 2-mL PourRite Ampules	each	2484600
Flask, volumetric, 50 mL	each	1457441
Flask, volumetric, 100 mL	each	1457442
Gloves, chemical resistant, size 10 <sup>1</sup>	each	2410105
Goggles, safety, standard	each	2927902
Notebook, field	each	2091800
Pipet, variable volume, 0.2–1.0 mL	each	BBP078
Pipet Tips, for BBP078 pipet	100/pkg	BBP079
Wipes, disposable, 28 x 37 cm	188/pkg	2932800

<sup>&</sup>lt;sup>1</sup> Additional sizes available

### G. Routine verification of the calibration for online chlorine analyzers

EPA Method 334.0 reference: Routine procedures section—online analyzers, paragraph 11.2

**Scope and application:** This procedure uses the grab sample method to verify the accuracy of online chlorine analyzers during normal operation. The results of the grab sample measurements must be within  $\pm 0.1$  mg/L or  $\pm 15\%$  (whichever is greater) of the online analyzer value.

### Before starting the test:

The maximum time between grab sample measurements must not exceed seven days.

The grab sample should be taken as close to the analyzer as possible.

Immediately analyze the collected grab sample.

DPD Free Chlorine Reagent Powder Pillows or DPD Total Chlorine Reagent Powder Pillows for a 25-mL sample size are used.

Use separate cells for DPD Free and DPD Total Chlorine testing to prevent cross-contamination.

All data from the grab sample and analyzer comparison must be recorded and maintained according to the requirements of the primacy agency.

### Test procedure

- Power on the instrument and select the program number or channel for the 0–4 mg/L DPD chlorine method.
- 2. Rinse the glass sample container several times with the sample water and collect approximately 50 mL of sample.
- 3. Rinse a clean sample cell two times with the sample water and then fill to the 10-mL mark.
- 4. Insert the sample cell into the instrument and push ZERO. The display shows 0.00 mg/L Cl<sub>2</sub>.
- **5.** Remove the sample cell and add the contents of 1 DPD Free Chlorine or 1 DPD Total Chlorine Powder Pillow. Cap and shake gently to mix.
- **6.** Insert the prepared sample into the instrument. Read the result at the time that is specified in the instrument test procedure.
- 7. Record the chlorine value of the grab sample. Compare this value to the value from the online analyzer. The two values should be within  $\pm 0.1$  mg/L or  $\pm 15\%$  of each other.
- 8. If the measured value of the grab sample differs from the measured value of the online analyzer by more than ±0.1 mg/L or ±15%, the problem must be corrected. Refer to Troubleshooting disagreement between the grab sample and analyzer measurements.
- **9.** Record the grab sample and analyzer comparison data. Maintain the data according to the requirements of the primacy agency.

Troubleshooting disagreement between the grab sample and analyzer measurements If the measurements from Routine verification of the calibration for online chlorine analyzers on page 12 do not agree, complete the following tasks to correct the problem.

- 1. Collect a second grab sample and repeat the analysis to make sure the results are consistent.
- 2. Complete analyzer maintenance (flow adjustment, pH adjustment, cleaning, new membrane or fresh reagents, etc.) to make sure there are no problems with the analyzer.
- 3. For remote or field sites, use the DPD Chlorine-MR Spec Check Secondary Standards to make sure that the grab sample instrument response is accurate (section B on page 5).

**Note:** If secondary standards were used to justify an analyzer calibration adjustment, complete the Routine verification of the calibration used for grab samples on page 11 within 24 hours with the same lot of reagents.

- **4.** Verify the accuracy of the grab sample measurement with a chlorine check standard solution. Refer to Routine verification of the calibration used for grab samples on page 11.
- 5. If the accuracy of the grab sample was verified, adjust the calibration curve of the analyzer to match the grab sample chlorine value. Follow the user instructions for the online chlorine analyzer to adjust the analyzer calibration.
- **6.** Collect another grab sample and measure the chlorine concentration to make sure that the analyzer and grab sample measurement agree.
- 7. Repeat steps 5 and 6 until the online analyzer measurements agree with the grab sample measurements.
- **8.** After one day of operation, collect and measure an additional grab sample to make sure that the analyzer performance is accurate.
- **9.** Return to the routine schedule for grab sample comparisons. A grab sample must be analyzed at least once each week.

### Replacement parts and reagents

### Reagents

Description	Unit	Catalog number
DPD Free Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1407099
or		
DPD Total Chlorine Reagent Powder Pillows for 25-mL samples	100/pkg	1406499
Chlorine Standard Solution, 2-mL PourRite® Ampule, 50–75 mg/L	20/pkg	1426820
or		
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	1426810
DPD Chlorine-MR Spec Check Secondary Standards Kit	each	2980500
Organic-free water	500 mL	2641549
Water, deionized	4 L	27256

### **Apparatus**

Description	Unit	Catalog number
Ampule Breaker Kit, 10-mL Voluette Ampules	each	2196800
Ampule Breaker, 2-mL PourRite Ampules	each	2484600
Beaker, 100 mL, glass	each	50042H
Flask, volumetric, 50 mL	each	1457441
Flask, volumetric, 100 mL	each	1457442
Gloves, chemical resistant, size 10 <sup>1</sup>	each	2410105
Goggles, safety, standard	each	2927902
Notebook, field	each	2091800
Pipet, variable volume, 0.2–1.0 mL	each	BBP078
Pipet Tips, for BBP078 pipet	100/pkg	BBP079
Wipes, disposable, 28 x 37 cm	188/pkg	2932800

<sup>&</sup>lt;sup>1</sup> Additional sizes available

### INITIAL DEMONSTRATION OF CAPABILITY - TOTAL RESIDUAL CHLORINE

### EPA Standard Method: 334.0

Each field sampler or person responsible for verification of chlorine analyzers must first complete an initial demonstration of capability (IDC) before the grab sample method can be used to verify the accuracy of online chlorine analyzers. The IDC requires both an accuracy and precision calculation. **Five replicates of a method blank** and **five replicates of a mid-range chlorine standard** are run. The average concentration of the five replicates of the mid-range chlorine standard must be within ±15% of the expected chlorine concentration. The average value of the five method blank values must be equal to or less than 1/3 the value of the lowest chlorine concentration used to initially verify the grab sample instrument. The precision or scatter in the concentration values of the five chlorine standard replicates must have a relative standard deviation (RSD) equal to or less than 15% of the average value.

Demonstration Date:	Analyst/Operator:		
Monitoring Device: Hach Pocket Colorimeter II DP	D Chlorine Pillow Lot#:	Expiration:	
Has the colorimeter been calibrated/checked anr	nually? Yes / No		

### Method Blank and Replicates:

- 1. Rinse a clean sample cell two times with organic-free water and then fill to the 10-mL mark.
- 2. Insert the sample cell into the instrument and push ZERO. The display shows 0.00 mg/L Cl2.
- 3. Remove the sample cell and add the contents of 1 DPD Total Chlorine Powder Pillow. Cap and shake gently to mix.
- 4. Insert the prepared method blank into the instrument. Read the value at the time that is specified in the instrument test procedure.
- 5. Record the value and dispose of the solution.
- Repeat steps 1–5 four additional times with the same sample cell to obtain the five replicate method blank values.
- 7. Calculate the accuracy and precision. Refer to the below example of how to Calculate the accuracy and precision.

Method Blank	ZERO	Add DPD	Time DPD Added	Reading (mg/L)	Time Read
Replicate 1	Y/N	Y/N			
Replicate 2	Y/N	Y/N			
Replicate 3	Y/N	Y/N			
Replicate 4	Y/N	Y/N			
Replicate 5	Y/N	Y/N			
Average	-	_	-		-

### **Test Procedure- Chlorine Standard Accuracy:**

- Calculate the volume of 25-30 mg/L chlorine standard that is necessary to make an approximate 2 mg/L Cl<sub>2</sub> standard solution. Approximately 100 mL of the diluted chlorine standard solution is required to complete the precision study. Refer to the below example of How to calculate the concentration of chlorine standard solutions.
- 2. Add approximately 25mL of organic-free water to a clean 100 mL volumetric flask.
- 3. Open a Chlorine Standard Solution Ampule, 25-30 mg/L Cl2.
- 4. Pipette the volume of the chlorine standard solution that was calculated in step 1 into the flask.
- 5. Fill the flask to the mark with organic-free water, stopper and gently invert several times to mix.
- 6. Immediately rinse a clean sample cell two times with a small portion of the diluted chlorine standard. Fill the sample cell to the 10-mL mark with the diluted standard.
- Insert the sample cell into the instrument and push ZERO. The display shows 0.00 mg/L Cl2.
- 8. Remove the sample cell and add the contents of 1 DPD Free Chlorine or 1 DPD Total Chlorine Powder Pillow for 10 mL samples. Cap and shake gently to mix.
- 9. Insert the prepared standard into the instrument. Read the result at the time that is specified in the instrument test procedure.
- 10. Record the value and dispose of the solution.
- 11. Repeat steps 6–10 four additional times with the same sample cell to obtain the 5 replicate mid-range chlorine standard values.
- 12. Calculate the accuracy and precision. Refer to the below example of how to Calculate the accuracy and precision.

### INITIAL DEMONSTRATION OF CAPABILITY - TOTAL RESIDUAL CHLORINE

Test Procedure	Standard Solution Value	ZERO	Add DPD	Time DPD Added	Reading (mg/L)	Time Read
Test 1		Y/N	Y/N			
Test 2		Y/N	Y/N			
Test 3		Y/N	Y/N			
Test 4		Y/N	Y/N			
Test 5		Y/N	Y/N			
Average		-	-	-		-

### How to calculate the concentration of chlorine standard solutions:

Chlorine standard solutions are prepared by dilution of a concentrated (50 to 75 mg/L Cl<sub>2</sub>) chlorine standard contained in ampules. The actual concentration changes for each lot of standard and is printed on the package label.

1. Calculate the volume of standard solution to be pipetted from the ampule:

<u>Desired final chlorine concentration x Volumetric flask volume</u> = Estimated pipetted volume Ampule standard concentration (from label)

- 2. Round the estimated volume to the nearest 0.05 mL (most pipettors read to 0.05 mL only)
- 3. Calculate the actual concentration of the diluted standard solution:

<u>Actual pipet volume x Ampule standard concentration (from label)</u> = Diluted standard concentration Volumetric flask volume

### Calculate the accuracy and precision:

### Accuracy:

Average (X) = Sum of 5 replicate values

5

Average (Method Blank) = Sum of 5 replicate method blank values

5

### Precision:

- 1. Use a spreadsheet application or a calculator function to determine the standard deviation (S) of the five replicate chlorine standard values.
- 2. Calculate the Relative Standard Deviation (RSD):

$$RSD = \underbrace{S}_{X} \times 100\%$$

Where:

S= the standard deviation for the 5 replicate standards

X= the average value of the 5 replicate chlorine standards

Demonstration	acceptabl	le? Yes / No
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Comments:	 	
Analyst/Operator Signature:	 	
Quality Control Officer Signature:		

### **Attachment E**

Manufacturer's Instructions

## CHLORIDE TEST KIT Model 8-P

Cat. No. 1440-01



# High Range, 0-400 mg/L Chloride

- Fill the plastic measuring tube level full with the water that is to be tested. Pour it into the mixing bottle.
- Add the contents of one Chloride 2 Indicator Powder Pillow. Swirl to mix as shown in Figure 1.
- Add the Silver Nitrate Titrant drop by drop to the water in the mixing bottle. Hold added. Count each drop as it is added until the water changes from yellow to orange in color. (An orange - red, rust color indicates the end point has been the dropper in a vertical position and swirl the bottle to mix after each drop is က

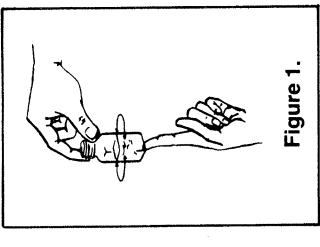
safety of the user if inappropriately handled. Please read all warnings before WARNING: The chemicals in this kit may be hazardous to the health and performing the test and use appropriate safety equipment.

TELEPHONE: WITHIN U.S. 800-227-4224, OUTSIDE U.S. 970-669-3050, TELEX: 160840 HACH COMPANY, P.O. BOX 389, LOVELAND, COLORADO 80359

- To obtain the chloride content of the water in mg/L as CI multiply the number of drops that were added by 20. 4
- To express the results as mg/L sodium chloride NaCl, multiply the mg/L chloride found in the test by 1.6. Ŋ.

# Low Range, 0-100 mg/L Chloride

- 1. Fill the mixing bottle to the 23-mL mark with the water that is to be tested.
- Add the contents of one Chloride 2 Indicator Powder Pillow. Swirl to mix as shown in Figure 1.
- Add the Silver Nitrate Titrant drop by drop to the water in the mixing bottle. Hold the dropper in a vertical position and swirl the bottle to mix after each drop is added. Count each drop as it is added until the water changes from yellow to orange in color. (An orange red, rust color indicates the end point has been exceeded.) ო
- by multiplying the number of drops that were added by 5. The chloride content of the water in mg/L as Cl is found 4
- (NaCI), multiply the mg/L chloride found in the test To express the results as mg/L sodium chloride Ŋ.



## REPLACEMENTS

Unit	pk/100	. 118 mL(4oz)MDB*	each	pk/6	each		118mL
Description	Chloride 2 Indicator Powder Pillows	Silver Nitrate Titrant, 0.0493 <i>N</i>	Measuring Tube	Mixing Bottle, with 23-mL mark	Clippers, small	Sodium Chloride Standard Solution, 298 mg/L as Cl,	491 mg/L as NaCl (not included in test kit)
Cat. No.	1043-99	23498-37	438-00	2327-06	936-00	14400-14	

\*Marked Dropping Bottle

It is suggested that reagent accuracy be checked from time to time by using a reliable standard, such as the Sodium Chloride Standard Solution, Cat. No. 14400-14 listed under Replacements. Follow regular instructions using the standard solution instead of a sample.

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10/95

DOC316.53.01475

## **TPH (Total Petroleum Hydrocarbons)**

Immunoassay<sup>1</sup> Method 10050

Scope and application: For water.

<sup>1</sup> This test is semi-quantitative. Results are shown as more or less than the threshold value used.



## **Test preparation**

## Before starting

This method analyzes for TPH in water samples. The test requires about 20 to 30 minutes for complete analysis. A maximum of 10 tests can be prepared at the same time.

Before the procedure starts, read the full procedure. Identify and prepare all the necessary reagents, cuvettes and other apparatus, then start the procedure.

Timing is very important in this procedure. Follow the instructions carefully.

It is very important to use a consistent technique to mix the solution in the cuvettes. Refer to Use of the 1-cm MicroCuvette rack on page 5. If the cuvettes are individually mixed, the results can be less consistent.

Be careful with the cuvettes. A scratch on the inner or outer cuvette surfaces can cause incorrect results. Carefully clean the outer surfaces with a clean, absorbent cloth or tissue before use.

Antibody cuvettes and enzyme conjugate are made in matched lots. Do not mix reagent lots.

Keep the color developing solution out of direct sunlight to prevent deterioration.

The cuvette rack can be inverted with the cuvettes in the rack. This lets the user prepare many samples at the same time. The cuvettes stay in the rack until the results are read in the instrument.

The recommended temperature for reagent storage is 4 °C (39.2 °F). Let the reagent temperature increase to room temperature before analysis.

Each reagent set has 20 antibody cuvettes. Use one antibody cuvette for each calibrator and each sample. Cuvettes are not reusable.

Use protective nitrile gloves for this procedure.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

### Items to collect

Description	Quantity
TPH Reagent Set	1
Caps, flip spout	1
Cylinder, graduated 10-mL	1
Marker, laboratory	1
Pipet, TenSette, 0.1–1.0 mL	
Pipet tips, for TenSette Pipet, 0.1–1.0-mL	1
Rack, for 1-cm Micro Cuvettes	1
Water, deionized	varies
Wipes, disposable	1
Wiretrol pipet	1

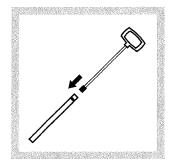
Refer to Consumables and replacement items on page 8 for order information.

## Sample collection and storage

- Analyze the samples as soon as possible for best results.
- If sample storage is necessary, collect the samples in glass or Teflon® containers. Clean the containers with soap and water, then rinse the containers with methanol. Use Teflon-lined caps for the containers. If Teflon-lined caps are not available, use aluminum foil as a substitute cap liner. Rinse the aluminum foil with methanol before use.
- Fill the container until it is full (no head space) and cover the container with a tightlysealed lid immediately after collection.
- Keep water samples in storage for no longer than 24 hours. Put the sample in an ice bath or a refrigerator to limit the loss of volatile compounds.

## **Use of the Wiretrol Pipet**

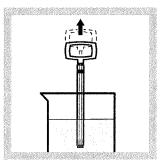
The Wiretrol Pipet accurately measures small quantities of liquids. The Wiretrol Pipet has two parts: a Teflon®-tipped plunger and a calibrated capillary tube. The plunger can be used many times. Discard the capillary tubes after one use.



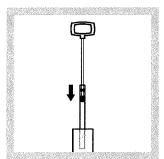
1. Make sure that the plunger tip is wet with the liquid. Carefully insert the plunger tip into the end of the capillary tube with the colored band.



2. Push the plunger tip to the other end of the capillary tube. Stop when the plunger tip barely extends beyond the end of the capillary tube.



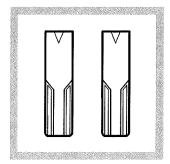
3. Insert the capillary tube below the surface of the liquid. Slowly and smoothly, pull the plunger up until the bottom of the plunger tip reaches the applicable volume line. Touch the end of the tube to the side of the vessel to release drops that remain on the capillary tube tip.



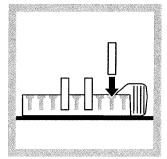
4. To release the liquid, insert the tip of the capillary tube below the surface of the receiving solution, and push the plunger downward in one smooth motion.

Change capillary tubes for each calibrator and sample.

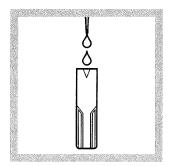
## Immunoassay procedure



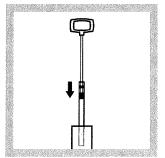
1. Put marks on the cuvettes to identify the samples and calibrators. Select the calibrator concentrations that are applicable to the expected sample concentration.



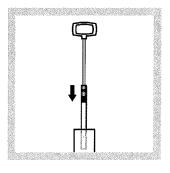
2. Insert the cuvettes into the rack. Make sure that the cuvettes are secure. Do not use force to put them into position because the cuvettes can spill or can be difficult to remove.



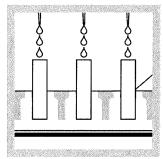
3. Use a pipet to add 0.5 mL of each water sample into a sample cuvette. Use a new pipet for each sample.



4. Use the Wiretrol pipet to add 50 μL of each calibrator to the applicable calibrator cuvette. Mix the cuvettes after each addition. Use a separate capillary tube for each solution. Have the necessary apparatus ready for this step and the next four steps. Do not wait—do these steps quickly.



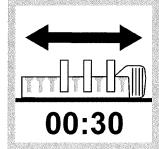
5. Use a Wiretrol pipet to add 50  $\mu$ L of methanol into each sample cuvette. Mix the contents of the cuvettes after each addition.



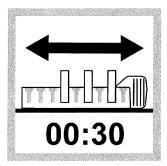
**6.** Immediately use a pipet to add 0.5 mL of TPH Enzyme Conjugate into each calibrator and sample cuvette. The same pipette tip can be used for this step.



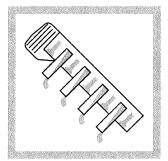
7. Set and start a timer for 10 minutes. A 10-minute reaction time starts.



8. Immediately mix the cuvettes for 30 seconds. Refer to Use of the 1-cm MicroCuvette rack on page 5 for the correct mixing procedure.



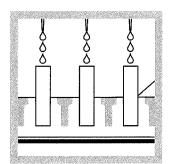
**9. After 5 minutes**, mix the contents of the rack a second time for 30 seconds.



**10.** At the end of the 10-minute reaction period, discard the contents of all the cuvettes into a waste container for disposal.



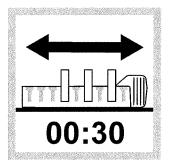
11. Fully rinse each cuvette with deionized water four times. Discard the contents into the waste container for disposal. Turn the cuvettes and rack upside down on a paper towel to dry. Carefully tap the cuvettes on the towel to remove the liquid.



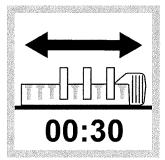
12. Start color development: Timing is very important. Make sure that the cuvettes are still in position in the rack. Use the pipet to add 0.5 mL of Color Developing Solution into each Antibody Cuvette. Use a new pipette tip for each cuvette.



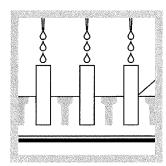
**13.** Set and start a timer for 10 minutes. A 10-minute reaction time starts.



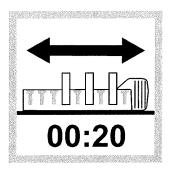
**14.** Immediately mix the cuvettes for 30 seconds.



**15. After 5 minutes**, mix the contents of the rack a second time for 30 seconds.



16. When the timer expires, use a pipette to add 0.5 mL of Stop Solution into each cuvette with the same pipette tip. Consistent technique is very important. Add the solution in the same sequence that was used for the Color Developing Solution addition.

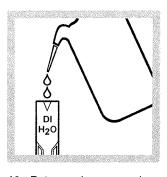


**17.** Slide the rack back and forth for 20 seconds. The blue solution color changes to yellow.

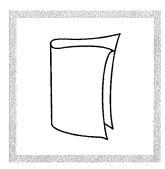


**18.** Set the instrument to channel 1 or channel 2. Refer to the instrument documentation.

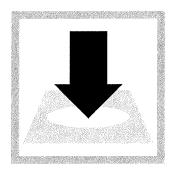
Make sure that the channel selected does not have a user-entered calibration.



**19.** Put a mark on a zeroing cuvette to identify it as the blank. Fill the cuvette with deionized water.



20. Clean all of the cuvettes.



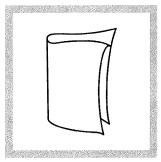
21. Insert the blank into the cell holder. Point the arrow mark on the cuvette toward the keypad.



**22.** Install the instrument cap over the cell holder.



23. Push ZERO. The display shows "0.000".



**24.** Clean the cuvette that contains the first calibrator.



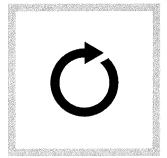
**25.** Insert the first calibrator into the cell holder. Point the arrow mark on the cuvette toward the keypad.



**26.** Install the instrument cap over the cell holder.



**27.** Push READ. Results show in absorbance units. Record the results.



**28.** Read the absorbance values of the remaining calibrators and samples. Record the results. Refer to Interpret and report results on page 6.

## Interferences

Interfering substance	Interference level
	Interferes above 2 ppm. To remove chlorine from the sample, add 1 drop of 0.1 N sodium thiosulfate per 100 mL of sample.

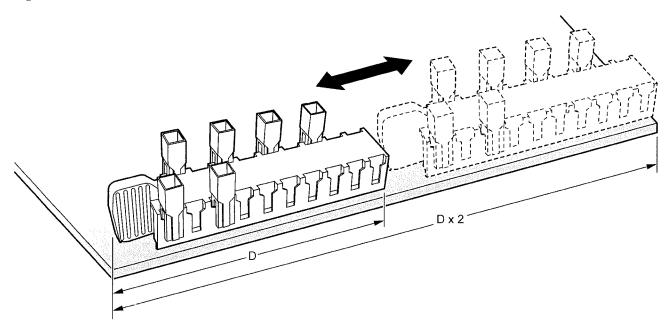
## Use of the 1-cm MicroCuvette rack

Use the MicroCuvette rack to get accurate and precise results for the immunoassay procedure during the analysis of several samples at a time. Refer to Figure 1.

**Insert the cuvettes in the rack**—Use the MicroCuvette rack to securely hold cuvettes that are set in the rack. Before the procedure starts, identify each cuvette with a sample or a calibrator number. Correctly insert the cuvettes in the rack. Do not force the cuvettes into the rack because the sample can spill or the cuvettes can be difficult to remove. The cuvettes must stay in position if the rack is inverted and carefully tapped.

**Mix the sample**—Put the rack on a hard, flat surface that is at least twice the length of the rack. Refer to Figure 1. Hold one end of the rack, then vigorously slide the rack back and forth along its axis for 30 seconds. The rack moves through a distance equal to its own length in each direction.

Figure 1 MicroCuvette rack



## Interpret and report results

There is an inverse relationship between the concentration of TPH and the absorbance reading. In other words, the higher the reading, the lower the concentration of TPH. Refer to Table 1.

Table 1 Relative TPH concentration

If the sample absorbance reading is	then the sample concentration is
Smaller than the calibrator reading	Larger than the calibrator reading
Larger than the calibrator reading	Smaller than the calibrator reading

## For example, if the readings are:

- TPH Calibrator 1 (20 ppm as diesel fuel): 0.480 Abs
- TPH Calibrator 2 (50 ppm as diesel fuel): 0.360 Abs
  - Sample 1: 0.200 Abs
  - Sample 2: 0.400 Abs
  - Sample 3: 0.550 Abs

### The interpretation for a sample:

- Sample 1: The sample reading is smaller than the readings for both calibrators. The sample concentration of in the sample is larger than 5 ppm diesel fuel.
- Sample 2: The sample reading is between the readings for the calibrators. The sample concentration of TPH is between 2 and 5 ppm diesel fuel.
- Sample 3: The sample reading is larger than the readings for both calibrators. The sample concentration of TPH is smaller than 2 ppm diesel fuel.

## Reagent storage and handling

- 1. Always wear gloves and eyewear for protection.
- 2. For long-term storage, make sure that the reagents are not in direct sunlight. Keep the reagent set at 4 °C (39.2 °F) when not in use. Warm the reagents to room temperature before use.

- 3. When not in use, seal the foil pouch that contains the antibody cuvettes.
- **4.** If the Stop Solution is in contact with the eyes, rinse fully for 15 minutes with cold water and get immediate medical help.

## Sensitivity

The antibodies used in the TPH Test Kit react with a variety of compounds found in petroleum fuels. Each TPH calibrator is formulated to show a known concentration of diesel fuel. Refer to Table 2 to use calibrators for other TPH compounds.

For example, to use the TPH calibrators for gasoline, find "Gasoline" in the correct table column. Then, read across the row to find the ppm of that hydrocarbon for each calibrator. For gasoline, TPH calibrator 1 = 1.5 ppm, TPH calibrator 2 = 3.5 ppm, etc.

TPH calibrator TPH calibrator TPH calibrator TPH calibrator Compound 1 (ppm) 2 (ppm) 3 (ppm) 4 (ppm) Diesel fuel 2 5 10 20 4 Gasoline 1.5 3.5 14 Kerosene 3.5 7.5 14 24 2 Benzene 4.5 8.5 16 3 5 9 Toluene 1.5 0.5 1.5 3.5 7.5 Ethylbenzene 2 7 m-Xylene 3.5 0.9 2 o-Xylene 1 8

0.5

1.5

Table 2 TPH compounds in water

## Dilute water samples

p-Xylene

**BTEX** 

0.3

0.5

For higher levels of TPH in water than those shown in Table 2 on page 7, dilute the sample with deionized water. To dilute a sample, refer to Table 3, then add that sample volume to a graduated cylinder and dilute to 50 mL with deionized water. Do the test. Refer to Table 2 on page 7 again to multiply the calibrator levels by the dilution multiplier.

0.9

2.5

16

4.5

For example, if a 0.5 mL water sample is diluted to 50 mL, the calibrator levels in Table 2 on page 7 for diesel fuel are approximately 200, 500, 1000 and 2000 ppm.

 mL sample
 Dilution multiplier

 0.5
 100

 1.0
 50

 2.0
 25

 5.0
 10

 10.0
 5

 25.0
 2

**Table 3 Dilution multipliers** 

## Summary of method

This method is the semi-quantitative screening for TPH based on thresholds as diesel fuel in the concentrations 2, 5, 10, 20 ppm as diesel fuel.

Immunoassay tests use antigen/antibody reactions to detect specific organic compounds in water. The walls of plastic cuvettes are layered with antibodies that are specific for atrazine. The antibodies selectively remove atrazine from complex sample matrices. A

prepared sample and a reagent with enzyme-conjugate molecules (analyte molecules attached to molecules of an enzyme) are added to the Antibody Cuvettes. During incubation, enzyme-conjugate molecules and atrazine compete for binding sites on the antibodies. Samples with higher levels of analyte have more antibody sites occupied by the analyte and fewer antibody sites occupied by the enzyme-conjugate molecules.

After incubation, the sample and unbound enzyme conjugate are rinsed from the cuvette and a color-development reagent is added. The enzyme in the conjugate catalyzes the development of color. Thus, there is an inverse relationship between color intensity and the amount of atrazine in the sample. The resulting color is then compared with a calibrator to determine if the analyte concentration in the sample is larger or smaller than the threshold levels. The atrazine concentration is inversely proportional to the color development-the lighter the color, the higher the atrazine concentration. The test results are measured at 450 nm.

## Consumables and replacement items

## Required reagents

Description	Quantity/Test	Unit	Item no.
Soil Extraction Kit	1	each	2775100
TPH Reagent Set	1	20 cuvettes	2774300
Water, deionized	varies	500 mL	27248

### Required apparatus

Description		Unit	Item no.
Caps, flip spout (for 500-mL deionized water bottle)	1	2/pkg	2581802
Marker, laboratory	1	each	2092000
Gloves, nitrile, medium	1	100/pkg	2550502
Pipet, TenSette <sup>®</sup> , 0.1–1.0 mL	1	each	1970001
Pipet Tips, for TenSette® Pipet, 0.1–1.0 mL	2	50/pkg	2185696
Pipet, Wiretrol <sup>®</sup> , 10–50 μL	1	each	2852200
Pipet, Wiretrol®, 50–1000 µL	1	each	2568905
Rack, for 1-cm Micro Cuvettes	1	each	4879900
Safety goggles, vented	1	each	2550700
Timer, talking	1	each	2764400
Wipes, disposable	1	280/pkg	2097000

## Optional reagents and apparatus

Description	Unit	Item no.
Graduated cylinder, 10-mL	each	108138
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB	32332



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING: In the U.S.A. – Call toll-free 800-227-4224

Outside the U.S.A. – Contact the HACH office or distributor serving you.

On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

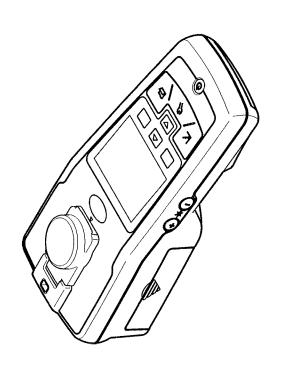
HACH COMPANY WORLD HEADQUARTERS Telephone: (970) 669-3050 FAX: (970) 669-2932

## DOC022.97.80041

# 2100Q and 2100Qis

04/2013, Edition 2





基本用户手册 基本取扱説明書 기본 사용 설명서

Manuel d'utilisation de base

**Basic User Manual** 

Manual básico del usuario Manual Básico do Usuário

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## Specifications

Specifications are subject to change without notice.

Specification	Details
Measurement method	Ratio turbidimetric determination using a primary nephelometric light scatter signal (90°) to the transmitted light scatter signal.
Regulatory	<b>2100Q</b> : Meets EPA Method 180.1 <b>2100Qis</b> : Meets ISO 7027
Lamp source	2100Q: Tungsten filament lamp 2100Qis: Light-emitting diode (LED) at 860 nm
Range	0–1000 NTU (FNU)
Accuracy	±2% of reading plus stray light from 0–1000 NTU (FNU)
Repeatability	±1% of reading or 0.01 NTU (FNU), whichever is greater
Resolution	0.01 NTU on lowest range
Stray light	≤ 0.02 NTU (FNU)
Signal averaging	Selectable on or off
Detector	Silicon Photodiode
Reading modes	Normal (Push to Read), Signal Averaging or Rapidly Settling Turbidity™
Calibration options	Single step RapidCal™ for Low-Level Regulatory Reporting from 0–40 NTU (FNU)
	Full range calibration from 0–1000 NTU (FNU)  Calibration to degrees of turbidity
Calibration logger	Records the last 25 successful calibrations
Verification logger	Logs the last 250 successful verifications
Data logger	500 records

Specification	Details
Power requirement	AC 100–240 V, 50/60 Hz (with power or USB/power module)
	4 AA alkaline batteries
	Rechargeable NiMH (for use with USB/power module)
Operating	Temperature: 0 to 50 °C (32 to 122 °F)
conditions	Relative Humidity: 0–90% at 30 °C, 0–80% at 40 °C, 0–70% at 50 °C, noncondensing
Storage conditions	-40 to 60 °C (-40 to 140 °F), instrument only
Interface	Optional USB
Sample required	15 mL (0.5 oz.)
Sample cells	Round cells $60 \times 25 \text{ mm}$ (2.36 x 1 in.) borosilicate glass with screw caps
Dimensions	22.9 x 10.7 x 7.7 cm (9.0 x 4.2 x 3.0 in.)
Weight	530 g (1.17 lb) without batteries
	620 g (1.37 lb) with four AA alkaline batteries
Meter enclosure rating	IP67 (closed lid, battery and module compartment excluded)
Protection class	Power supply: Class II
Certification	CE certified
Warranty	1 year (EU: 2 years)

## **General information**

In no event will the manufacturer be liable for direct, indirect, special, incidental or consequential damages resulting from any defect or omission in this manual. The manufacturer reserves the right to make changes in this manual and the products it describes at any time, without notice or obligation. Revised editions are found on the manufacturer's website.

## Safety information

## NOTICE

The manufacturer is not responsible for any damages due to misapplication or misuse of this product including, without limitation, direct, incidental and consequential damages, and disclaims such damages to the full extent permitted under applicable law. The user is solely responsible to identify critical application risks and install appropriate mechanisms to protect processes during a possible equipment malfunction.

Please read this entire manual before unpacking, setting up or operating this equipment. Pay attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

Make sure that the protection provided by this equipment is not impaired. Do not use or install this equipment in any manner other than that specified in this manual.

## Use of hazard information

## A DANGER

Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

## **WARNING**

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

## **ACAUTION**

Indicates a potentially hazardous situation that may result in minor or moderate

## NOTICE

Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.

## Precautionary labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed. A symbol on the instrument is referenced in the manual with a precautionary statement.



This is the safety alert symbol. Obey all safety messages that follow this symbol to avoid potential injury. If on the instrument, refer to the instruction manual for operation or safety information.



This symbol indicates that a risk of electrical shock and/or electrocution exists.



Electrical equipment marked with this symbol may not be disposed of in European public disposal systems after 12 August of 2005. In conformity with European local and national regulations (EU Directive 2002/96/EC), European electrical equipment users must now return old or end-of-life equipment to the Producer for disposal at no charge to the liser.

Note: For return for recycling, please contact the equipment producer or supplier for instructions on how to return end-of-life equipment, producer-supplied electrical accessories, and all auxiliary items for proper disposal.

## Certification

Canadian Radio Interference-Causing Equipment Regulation, IECS-003, Class A:

Supporting test records reside with the manufacturer.

This Class A digital apparatus meets all requirements of the Canadian Interference-Causing Equipment Regulations.

Cet appareil numérique de classe A répond à toutes les exigences de la réglementation canadienne sur les équipements provoquant des interférences.

## FCC Part 15, Class "A" Limits

Supporting test records reside with the manufacturer. The device complies with Part 15 of the FCC Rules. Operation is subject to the following conditions:

1. The equipment may not cause harmful interference.

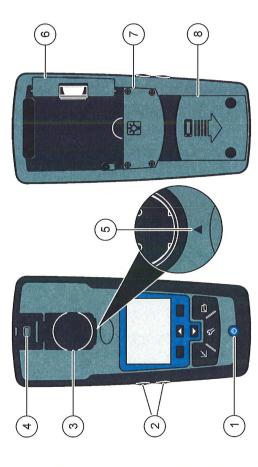
The equipment must accept any interference received, including interference that may cause undesired operation. Changes or modifications to this equipment not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment. This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference, in which case the user will be required to correct the interference at their expense. The following techniques can be used to reduce interference problems:

- Disconnect the equipment from its power source to verify that it is or is not the source of the interference.
- If the equipment is connected to the same outlet as the device experiencing interference, connect the equipment to a different outlet.
- 3. Move the equipment away from the device receiving the interference.
- Reposition the receiving antenna for the device receiving the interference.
- Try combinations of the above.

## **Product overview**

The 2100Q and 2100Q*is* portable turbidimeters measure turbidity from 0 to 1000 NTU (FNU). Primarily for field use, the portable meter operates on four AA batteries. Data can be stored and transferred to a printer, computer or USB storage device.

Figure 1 Product overview

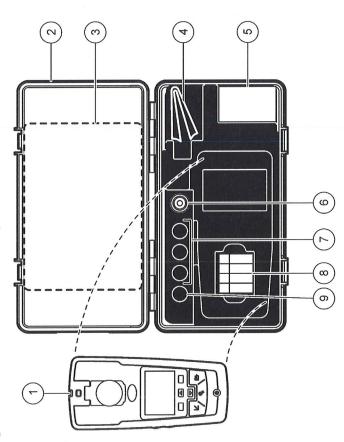


1 Power on or off5 Alignment arrow2 Backlight keys (+ and -)6 Module3 Sample cell holder with lid7 Lamp compartment4 Attachment for lanyard8 Battery compartment				
	~	Power on or off	2	Alignment arrow
	2	Backlight keys (+ and -)	9	Module
Attachment for lanyard	က	Sample cell holder with lid	7	Lamp compartment
	4	Attachment for lanyard	œ	Battery compartment

## Product components

Refer to Figure 2 to make sure that all components have been received. If any of these items are missing or damaged, contact the manufacturer or a sales representative immediately.

Figure 2 2100Q and 2100Qis components



•	_	2100Q or 2100Q <i>is</i> turbidimeter	9	6 Silicone oil
. 4	2	2 Carrying case	7	7 20, 100 and 800 NTU StablCal calibration standards
1.,	က	3 User manual and Quick reference guide	œ	8 AA alkaline batteries (pk/4)
7	4	4 Oiling cloth	6	9 StablCal 10 NTU verification
47	2	5 1" sample cell (10 mL) with cap (pk/6)		standard

## Installation

## Multiple

Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

**ACAUTION** 

## Install the battery

## AWARNIN



Explosion hazard. An expired battery can cause hydrogen gas buildup inside the instrument. Replace the battery before it expires. Do not store the instrument for long periods with a battery installed.

## **AWARNING**

Potential fire hazard. Use only alkaline or nickel metal hydride batteries (NiMH) in the meter. Other battery types or incorrect installation can cause a fire. Never mix battery types in the meter.

## MO TO V

The battery compartment is not waterproof. If the battery compartment becomes wet, remove and dry the batteries and dry the interior of the compartment. Check the battery contacts for corrosion and clean them if necessary.

## NOTICE

When using nickel metal hydride (NiMH) batteries, the battery icon will not indicate a full charge after freshly charged batteries have been inserted (NiMH batteries are 1.2 V versus 1.5 V for alkaline batteries). Even though the icon does not indicate complete charge, 2300 mAH NiMH batteries will achieve 90% of instrument operation lifetime (before recharge) versus new alkaline batteries.

## MOJICE MOJICE

To avoid potential damage to the meter from battery leakage, remove the meter batteries prior to extended periods of non-use.

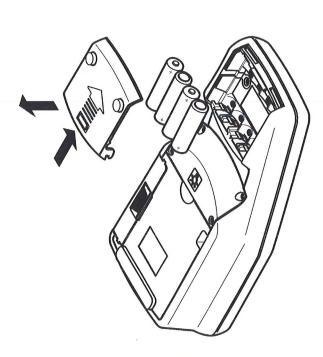
The meter can be powered with AA alkaline or rechargeable NiMH batteries. To conserve battery life, the meter will power off after 10 minutes of inactivity, the backlight powers off after 30 seconds. This time can be changed in the Power Management menu.

**Note**: Rechargeable batteries will only be recharged with the USB/power module. Refer to the module documentation for further information.

For battery installation refer to Figure 3.

- Remove the battery cover.
- 2. Install 4 AA alkaline or 4 AA nickel metal hydride (NiMH) batteries. Make sure that the batteries are installed in the correct orientation.
- 3. Replace the battery cover.

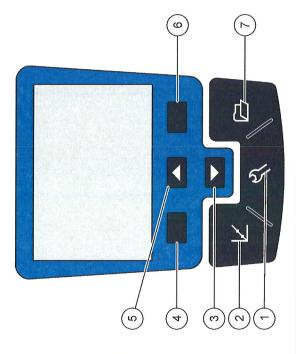
Figure 3 Battery installation



## User interface and navigation

## User interface

Figure 4 Keypad description

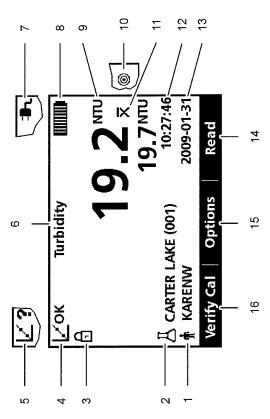


•				
	7	SETTINGS key: select menu     options for setting up the meter	ro	5 UP key: scroll through menus, enter numbers and letters
	2	2 CALIBRATION key: shows calibration screen, start calibration, select cal options	9	6 RIGHT key (contextual): read turbidity sample, selects or confirms options, opens/jumps to sub-menus
	က	3 DOWN key: scroll through menus, enter numbers and letters	7	DATA MANAGEMENT key: view, delete or transfer stored data
	4	4 LEFT key (contextual): access for calibration verification, cancels or exits the current menu screen to the previous menu screen		

## Display description

The measurement screen shows the turbidity, unit, calibration status, date and time, operator ID (if setup) and sample ID (if setup). Refer to Figure 5.

Figure 5 Single screen display



	1 Operator identification	6	9 NTU (Nephelometric Turbidity Unit) or FNU (Formazin Turbidity Unit)
7	2 Sample identification	5	10 Reading mode: Rapidly Settling Turbidity (Target icon)
က	3 Stability or display lock indicator	7	11 Reading mode: Signal Average (X-bar icon)
4	Calibration status indicator (Calibration OK=pass)	12	12 Time
5	5 Calibration status indicator (Calibration ?=fail)	13	13 Date
9	6 Parameter title	14	14 Read (contextual: OK, Select)
7	7 AC power icon	15	15 Options (contextual)
∞	8 Battery icon	16	16 Verification calibration

## Navigation

The meter contains a Settings menu, Reading Options menu, Calibration Options menu and Calibration Verification Options menu to change various options. Use the **UP** and **DOWN** keys to highlight different options. Push the **RIGHT** key to select an option. There are two ways to change options:

- Select an option from a list: Use the UP and DOWN keys to select an option. If check boxes are shown, more than one option can be selected. Push the LEFT key under Select.
- Note: To deselect check boxes, push the LEFT key under Deselect.
- Enter an option value using the arrow keys: Push the UP and DOWN keys to enter or change a value.
- 3. Push the RIGHT key to advance to the next space.
- 4. Push the RIGHT key under OK to accept the value.

## Startup

## Turn the meter on and off

Dush the ON/OFF key to turn on or turn off the meter. If the meter does not turn on, make sure that the batteries, or the module, are properly installed or that the AC power supply is properly connected to an electrical outlet.

**Note:** The Auto-Shutoff option can also be used to turn off the meter. Additional information is available on the manufacturer's website.

## Change the language

There are three options to set the language:

- The display language is selected when the meter is powered on for the first time.
- The display language is selected when the power key is pushed and
- The language can be changed from the Settings menu.
- 1. Select a language from the list. Confirm with OK.
- 2. Push Done when the update is complete.

## Change the date and time

The date and time can be changed from the Date & Time menu.

- Push the SETTINGS key and select Date & Time.
- 2. Update the time and date information:

Option	Description
Format	Select one of the formats for the date and time:
	yyyy-mm-dd 24h

yyyy-mm-dd 12h

dd-mm-yyyy 24h

dd-mm-yyyy 12h

mm/dd/yyyy 24h mm/dd/yyyy 12h Enter the current date Date

Enter the current time Time

The current date and time will be shown on the display.

After the date and time setup, the meter is ready to take a reading.

## Standard operation

## Use a sample ID

The sample ID tag is used to associate readings with a particular sample location. If assigned, stored data will include this ID.

- Select Sample ID in the Settings menu.
- 2. Select, create or delete a sample ID:

Description	Select an ID from a list. The current ID will be	associated with sample data until a different ID is	selected.
Option	Current ID		

Create a New Sample Enter a name for a new sample ID. ID

Delete an existing sample ID. Delete Sample ID

Use an operator ID

The operator ID tag associates readings with an individual operator. All stored data will include this ID.

- 1. Select Operator ID in the Settings menu.
- 2. Select, create or delete an operator ID:

Description	Select an ID from a list. The current ID will be associated with sample data until a different ID is selected.
Option	Current ID

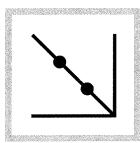
Enter a name for a new operator ID (maximum 10 names can be entered). Create a New Operator ID

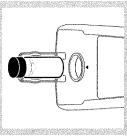
Delete an existing operator ID. Delete Operator ID

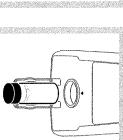
## Advanced operation

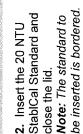
# Calibrate the turbidimeter with StablCal® Standards

Note: For best accuracy use the same sample cell or four matched sample cells for compartment so the diamond or orientation mark aligns with the raised orientation all readings during calibration. Insert the sample cell in the instrument cell mark in front of the cell compartment.









CALIBRATION key to

Push the

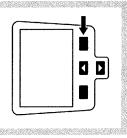
enter the Calibration

inserting the standard. each standard before

Note: Gently invert

display.

instructions on the mode. Follow the

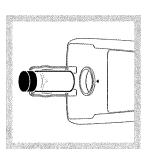


Standard.

complete a 2 point calibration.

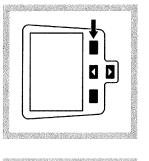
Stabilizing and then shows the result. 3. Push Read. The display shows

Reading modes



review the calibration 5. Push Done to

4. Repeat Step 2 and



Þ

Additional information is manufacturer's website. automatically goes into 6. Push Store to save the Verify Cal mode. complete, the meter After a calibration is available on the the results.

## details. 3 with the 100 NTU and Note: Push Done to 800 NTU StablCal

- Push the UP or DOWN key to enter the Reading Options menu.
- Select Reading Mode to select one of the following options:

Description Option The normal mode reads and averages three readings. The result is shown after the reading. Normal (Default setting)

Description	The City of the Ci
Option	Cicasi

Signal Average

The Signal Average mode compensates for reading fluctuations caused by drifting of sample particles through the light path.

The X-bar icon is shown on the display when signal averaging is on.

The Signal Average mode measures 12 times and starts to show the average after three readings. The final result is the average of all 12 readings.

Rapidly Settling Turbidity™ (RST)

The Rapidly Settling Turbidity (RST) mode calculates and continuously updates the turbidity reading of the sample to a confidence of 95%, based on the accumulated trend of the real time measured values.

The RST mode is best used on samples that settle rapidly and continuously change in value. The reading is based on a correctly prepared sample that is homogeneous at the beginning of the reading. It is best applied to samples that are greater than 20 NTU. The sample must be mixed thoroughly by inversion immediately before inserting it into the meter.

The target icon is shown on the display when the Rapidly Settling Turbidity is on.

The Rapidly Settling Turbidity reads and calculates five eadings while showing intermediate results.

## Maintenance



Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

**ACAUTION** 

## Clean the meter

The meter is designed to be maintenance-free and does not require regular cleaning for normal operation. Exterior surfaces of the meter may be cleaned as necessary.

Note: Do not clean the meter with solvents to avoid damaging the material.

1. Clean the meter with a dust- and lint-free dry or slightly damp cloth. A mild soap solution can also be used for liposoluble contamination.

## Apply silicone oil to a sample cell

Sample cells and caps must be extremely clean and free from significant scratches. Apply a thin coating of silicone oil on the outside of the sample cells to mask minor imperfections and scratches that may contribute to light scattering.

**Note:** Use only the provided silicone oil. This silicone oil has the same refractive index as the sample cell glass.



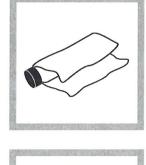
2. Apply a small bead of silicone oil from the top to the bottom of the

1. Clean the inside and

outside of the cells and caps by washing with a

rinses with distilled or demineralized water.

laboratory glass cleaning detergent. Follow with multiple



3. Use the provided oiling cloth to spread the oil uniformly. Wipe off the excess so that only a thin coat of oil is left. Make sure that the sample cell is almost dry with little or no visible oil.

Natible oil.

Note: Store the oiling cloth in a plastic storage bag to keep the cloth clean.

## Store the sample cells

## NOTICE

Do not air dry the sample cells.

Note: Always store the sample cells with caps on to prevent the cells from drying.

- Fill the sample cells with distilled or demineralized water.
- Cap and store the sample cells.
- . Wipe the outside of the sample cells dry with the a soft cloth.

## Replace the battery



Explosion hazard. An expired battery can cause hydrogen gas buildup inside the instrument. Replace the battery before it expires. Do not store the instrument for long periods with a battery installed.

**AWARNING** 

## **AWARNING**

Potential fire hazard. Use only alkaline or nickel metal hydride batteries (NiMH) in the meter. Other battery types or incorrect installation can cause a fire. Never mix battery types in the meter.

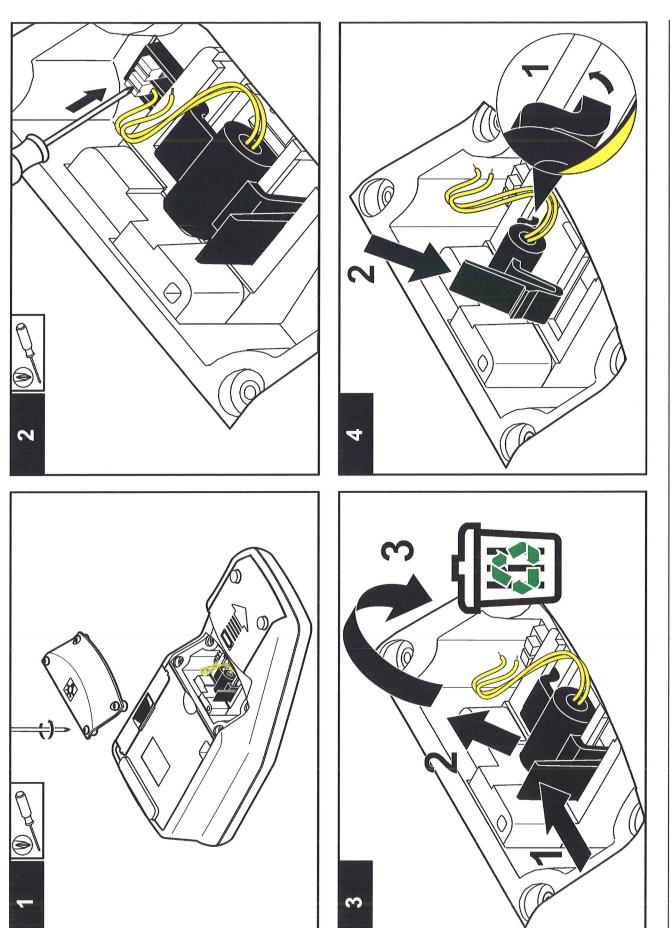
For battery replacement refer to Install the battery on page 6.

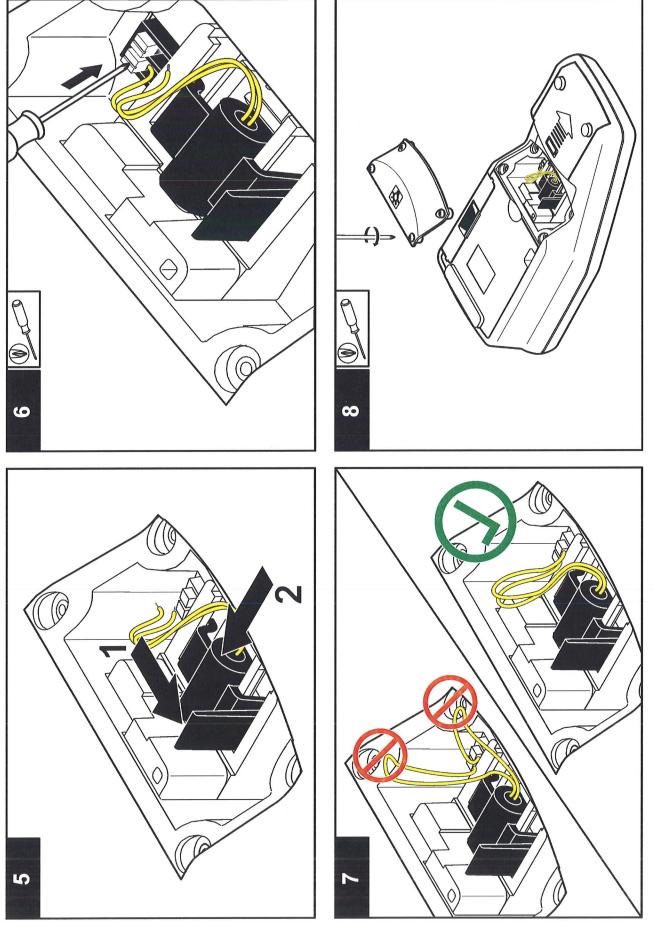
- Remove the battery cover.
- 2. Remove the batteries.
- **3.** Install 4 AA alkaline or 4 AA nickel metal hydride (NIMH) batteries. Make sure that the batteries are installed in the correct orientation.
- 4. Replace the battery cover.

## Replace the lamp

## **ACAUTION**

Burn Hazard. Wait until lamp cools down. Contact with the hot lamp can cause burns.





14 English

## **Troubleshooting**

Refer to the following tab

Refer to the following tak symptoms, possible cau:	Refer to the following table for common problem messages or symptoms, possible causes and corrective actions.	messages or is.
<b>Error/Warning</b>	Description	Solution
Close lid and push Read.	The lid is open or lid detection failed.	Make sure that the lid is closed during reading and re-read.
Low Battery!	Battery is low.	Insert new batteries     Connect USB/power     module if     rechargeable batteries     are used
ADC Failure!	Hardware error causing reading to fail.	Repeat the reading.
Detector signal too low!	Insufficient light on the 180° detector.	Check for obstructed light path.     Check the lamp.
Overrange!	Turbidity too high- caused probably by calibrating with RapidCal <sup>™</sup> only.	<ul> <li>Calibrate the upper range.</li> <li>Dilute the sample.</li> </ul>
Underrange!	The measured absorbance is below the calibration range.	Repeat calibration
Please check the lamp!	Signals are too low on the 90° and 180° detector.	2100Q: The lamp is defective. Change the lamp (refer to Replace the lamp on page 12). 2100Qis: Contact technical support.

ה			Error/Warning	Description	Solution
able	able for common problem messa uses and corrective actions.	messages or Is.	Temperature too high! Switch off instrument.	Temperature has exceeded the meter Imits (>60 °C or	Turn off the meter and let it cool down.
	Description	Solution		>140 °F).	
Þŏ	The lid is open or lid detection failed.	Make sure that the lid is closed during reading and re-read.	RST: Average value!	Solids are settling too slowly. The reading mode is not suitable for this sample.	Select Normal or Signal Average reading mode.
<u> </u>	Battery is low.	Insert new batteries     Connect USB/power     module if     rechargeable batteries     are used	Confidence level is < 95%	The reading mode Rapidly Settling Turbidity did not meet the range of ≥ 95% confidence.	Invert the sample several times so that the solids allocate. Repeat the reading again.
T E	Hardware error causing reading to fail.	Repeat the reading.			Switch to the Normal reading mode if the sample is stable and
<u> </u>	Insufficient light on the 180° detector.	Check for obstructed light path.			does not have settable solids.
		Check the lamp.	Standard value out of range. Insert standard	Used incorrect standard value for the reading.	Insert the appropriate standard and read again.
<u> </u>	Turbidity too high-	Calibrate the upper	and push Read		
225	caused probably by calibrating with RapidCal <sup>™</sup> only.	range.  • Dilute the sample.	ID already in use. Enter new ID	The Operator or Sample ID is unavailable as it is already assigned.	Create a new ID.
8 5 5 1	The measured absorbance is below the calibration range.	Repeat calibration	Error - Security Please set password before activating security	No password is created.	Create a new password.
σ≨ĕ	Signals are too low on the 90° and 180° detector.	2100Q: The lamp is defective. Change the lamp (refer to Replace the lamp on page 12)	Please enter at least one character.	Password must contain minimum of one character.	Create a password of at least one character.
		2100Qis: Contact technical support.	Password incorrect. Please retry.	Incorrect password was entered.	Enter the appropriate password.

<b>Error/Warning</b>	Description	Solution
Please disconnect the USB cable from your computer.	Data storage does not respond while connected to the meter and the computer.	Disconnect the USB cable from the meter and try sending data again.
USB module memory full. Delete data and try again.	Data storage is full.	<ol> <li>Connect USB/power module to the computer.</li> <li>Download the stored data to the computer.</li> <li>Delete Data Log on the module.</li> </ol>
Delete Last Reading Failed!	Error in the data storage.	Turn the meter off and on. If the error message
Delete Data Log failed!		still occurs, contact technical support.
Can't read data set!		
Can't store data!		
Can't store to the Reading Log!		
Can't store to the Verify Cal Log!		
Error storing data!		
Error reading data!		



## **MODEL 1200-NH • CODE 3680-01**

QUANTITY	CONTENTS	CODE
30 mL	Ammonia Nitrogen Reagent #1	V-4797-G
3 x 30 mL	*Ammonia Nitrogen Reagent #2	*V-4798-G
1	Pipet, 1 mL, plastic	0354
1	Colorimeter Tubes, with caps	0290-6
1	Water Sample Collecting Bottle	0688
1	1200 Colorimeter for Ammonia Nitrogen	26737

<sup>\*</sup>WARNING: Reagents marked with a \* are considered to be potential health hazards. To view or print a Material Safety Data Sheet (MSDS) for these reagents see MSDS CD or www.lamotte.com. To obtain a printed copy, contact LaMotte by e-mail, phone or fax.

To order individual reagents or test kit components, use the specified code number.

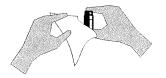
## INTRODUCTION

Ammonia nitrogen is present in various concentrations in many surface and ground water supplies. Any sudden change in the concentration of ammonia nitrogen in a water supply is cause for suspicion. A product of microbiological activity, ammonia nitrogen is sometimes accepted as chemical evidence of pollution when encountered in natural waters.

Ammonia is rapidly oxidized in natural water systems by special bacterial groups that produce nitrite and nitrate. This oxidation requires that dissolved oxygen be available in the water. Ammonia is an additional source of nitrogen as a nutrient which may contribute to the expanded growth of undesirable algae and other forms of plant growth that overload the natural system and cause pollution.

## AMMONIA NITROGEN TEST PROCEDURE: NESSLER METHOD

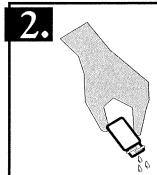
Read the 1200 Colorimeter Manual before proceeding. Carefully wipe tubes dry before inserting into the colorimeter chamber.



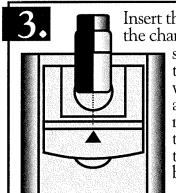
## **AMMONIA NITROGEN**



Fill the Water Sample Collecting Bottle (0688) with sample water. This will be used to dispense sample water for the tests.



Rinse and fill a colorimeter tube (0290) to the 10 mL line with sample water. Cap and wipe dry.



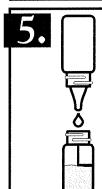
Insert the tube into the chamber, being sure to align the index lin

the index line with the arrow on the meter. Close the lid. This tube is the blank or zero.

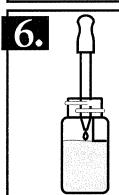


Push the **READ** button to turn the meter on. Press

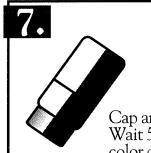
the **ZERO** button and hold it for 2 seconds until **BLR** is displayed. Release the button to take a blank reading (0.0 ppm).



Remove tube from colorimeter. Add 8 drops of Ammonia Nitrogen Reagent #1 (V-4797). Cap and mix.



Use 1.0 mL pipet (0354) to add 1.0 mL of \*Ammonia Nitrogen Reagent #2 (V-4798).



Cap and invert to mix. Wait 5 minutes for full color development. Wipe tube dry.



Align the index line with the arrow on the meter, insert tube

Close the lid. Push the **READ** button. Record results as ppm Ammonia Nitrogen (NH<sub>3</sub>-N).

into chamber.

**NOTE:** For the best possible results, carry a reagent blank through the procedure. After scanning the blank in Step 4, perform the test procedure on clear, colorless, distilled or deioized water. Subtract results of regent blank from all subsequent test results.

**NOTE:** If the reading displays *ER2*, repeat procedure on diluted sample, and multiply the result by the appropriate dilution factor. See 1200 Colorimeter Instruction Manual for procedure.

## **CALCULATIONS**

To express results as Unionized Ammonia (NH<sub>3</sub>):

Unionized Ammonia (NH<sub>3</sub>) = ppm Ammonia Nitrogen (NH<sub>3</sub>-N) x 1.2 To express results as Ionized Ammonia (NH<sub>4</sub><sup>+</sup>):

Ionized Ammonia ( $NH_4^+$ ) = ppm Ammonia Nitrogen ( $NH_3$ -N) x 1.3

Ammonia in water occurs in two forms: toxic unionized ammonia (NH<sub>3</sub>) and the relatively non-toxic ionized form, ammonium ion (NH<sub>4</sub> $^+$ ). This test method measures both forms as ammonia-nitrogen (NH<sub>4</sub>-N) to give the total ammonia-nitrogen concentration in water. The actual proportion of each compound depends on temperature, salinity, and pH. A greater concentration of unionized ammonia is present when the pH value and salinity increase.

- 1. Consult the table below to find the percentage that corresponds to the temperature, pH and salinity of the sample.
- 2. To express the test result as ppm Unionized Ammonia Nitrogen (NH<sub>3</sub>-N), multiply the total ammonia-nitrogen test result by the percentage from the table.
- 3. To express the test result as ppm Ionized Ammonia Nitrogen (NH<sub>4</sub><sup>+</sup>-N), subtract the unionized ammonia-nitrogen determined in Step 2 from the total ammonia nitrogen.

	10	)°C	15	5°C	20	°C	25	°C
рН	FW <sup>1</sup>	SW <sup>2</sup>	FW	SW	FW	SW	FW	SW
7.0	0.19		0.27		0.40		0.55	
7.1	0.23		0.34		0.50		0.70	
7.2	0.29		0.43		0.63		0.88	
7.3	0.37		0.54		0.79		1.10	
7.4	0.47		0.68		0.99		1.38	
7.5	0.59	0.459	0.85	0.665	1.24	0.963	1.73	1.39
7.6	0.74	0.577	1.07	0.836	1.56	1.21	2.17	1.75
7.7	0.92	0.726	1.35	1.05	1.96	1.52	2.72	2.19
7.8	1.16	0.912	1.69	1.32	2.45	1.90	3.39	2.74
7.9	1.46	1.15	2.12	1.66	3.06	2.39	4.24	3.43
8.0	1.83	1.44	2.65	2.07	3.83	2.98	5.28	4.28
8.1	2.29	1.80	3.32	2.60	4.77	3.73	6.55	5.32
8.2	2.86	2.26	4.14	3.25	5.94	4.65	8.11	6.61
8.3	3.58	2.83	5.16	4.06	7.36	5.78	10.00	8.18
8.4	4.46	3.54	6.41	5.05	9.09	7.17	12.27	10.10
8.5	5.55	4.41	7.98	6.28	11.18	8.87	14.97	12.40

<sup>&</sup>lt;sup>1</sup>Freshwater data from Trussel (1972).

<sup>&</sup>lt;sup>2</sup>Seawater values from Bower and Bidwell (1978). Salinity for the Seawater values = 34% at an ionic strength of 0.701 m.

## FOR EXAMPLE:

A fresh water sample at 20°C has a pH of 8.5 and the test result is 1.0 ppm as total Ammonia-Nitrogen.

- 1. The percentage from the table is 11.18% (or 0.1118).
- 2. 1 ppm total Ammonia-Nitrogen x 0.1118 = 0.1118 ppm Unionized Ammonia-Nitrogen
- Total Ammonia-Nitrogen
   Unionized Ammonia-Nitrogen
   Ionized Ammonia-Nitrogen
   0.1118 ppm
   0.8882 ppm

## AMMONIA NITROGEN TEST METHOD SPECIFICATIONS

## **APPLICATION**

Drinking, surface, and saline waters; domestic and industrial wastes.

## **RANGE**

0 to 5.0 ppm Ammonia Nitrogen

## **METHOD**

Ammonia forms a colored complex with Nessler's Reagent in proportion to the amount of ammonia present in the sample. Rochelle salt is added to prevent precipitation of calcium or magnesium in undistilled samples.

## HANDLING & PRESERVATION

Preservation is accomplished by the addition of 2 mL of concentrated H<sub>2</sub>SO<sub>4</sub> at 4°C.

## **INTERFERENCES**

Sample turbidity and color may interfere. Turbidity may be removed by a filtration procedure. Color interference may be eliminated by adjusting the instrument to 100%T with a sample blank.

## LaMOTTE COMPANY

Helping People Solve Analytical Challenges®
PO Box 329 • Chestertown • Maryland • 21620 • USA
800-344-3100 • 410-778-3100 (Outside U.S.A.) • Fax 410-778-6394
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## **Attachment F**

Golder-Dominion Purchase Order Agreement



## Purchase order

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PO number/date

70300442 / 03/04/2016

Contact person/Telephone

Kevin Brannan/804-273-3135

Our fax number

888-219-1940

GOLDER ASSOCIATES INC 2108 2108 W LABURNUM AVE SUITE 200 RICHMOND VA 23227

Your vendor number with us 300000861

Vendor Phone: 517 2854704 Vendor Fax: 804 3582900

> Your person responsible Ron DiFrancesco

Please deliver to:

Bremo Power Station 1038 Bremo Road

Bremo Bluff VA 23022

Valid from: Valid to:

03/04/2016 02/28/2019

Delivery date: 12/31/2018

Invoice To:

Dominion Accounts Payable PO Box 25459 Richmond, Va 23260-5459 If Taulia/Credit Card/ERS/EDI NOTE: Disregard

Sales Tax instructions

are contained on the last page of this document.

Contact Information:

https://www.dom.com/suppliers/active -suppliers/contact-information.jsp

Terms of delivery: FOB destination delivered

Terms of payment: EDI - Net 30 Days

Currency USD

### Acceptance:

Purchaser requires a signed acceptance copy of the signature page of this Purchase Order. It must be signed, dated and returned within 10 calendar days of receipt or acceptance to the Purchaser's Supply Chain representative listed above. Please e-mail a signed copy of this Purchase Order to kevin.e.brannan@dom.com

If the Supplier does not return a signed Acceptance Copy to the buyer for this Purchase Order, the Supplier will not receive any payments until the signed Acceptance Copy of this Purchase Order is returned to the buyer.

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 2 of 13 RICHMOND VA 23227

\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*

A current Certificate of Insurance must be provided prior to the commencement of any on site work. Please e-mail the certificate to kevin.e.brannan@dom.com

\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*

### General:

This is a Purchase Order (hereinafter also referred to as "Order" or "Agreement") between Virginia Electric and Power Company (hereinafter also referred to as "Purchaser") and Supplier as shown at top of the Order to provide Environmental Sampling services as specified in the scope of work.

Supplier Contact Information:

Name: Ron DiFrancesco Phone: 804-521-1775

Email: Ron\_DiFrancesco@golder.com

## Applicable Terms and Conditions:

Supplier shall perform and complete services described herein in accordance with:

- -This Purchase Order Document
- -The Terms and Conditions in Value Contract 46027533

### Order of Precedence:

In the event of any conflict among the following documents, or any ambiguity arises among two or more of the following documents, the provisions of the first listed document below shall control over all of the other documents listed in numerical order below it, and the provisions of the second listed document shall control over the documents as listed in numerical order below it, and so forth in the order in which the documents are numbered.

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 3 of 13 RICHMOND VA 23227

- 1. The terms of a duly authorized and executed change order or contract amendment with regard to the subject matter of the change order or contract amendment
- 2. The Terms and Conditions in Value Contract 46027533
- 3. This Purchase Order Document

### Method of Order Release:

Work authorized hereunder may be made by verbal or written release. Authority to release services is limited to Purchaser's Supply Chain Management (SCM) personnel and the individuals delineated in the "Authorized Station Contacts" section below.

### Supplier Safety Rates:

The Supplier is responsible for promptly reporting, to the Purchaser contact identified below, any changes in the Supplier's OSHA Incident Rate, Experience Modification Rate (EMR) or the receipt of any OSHA citations during the Term of this Agreement. Failure to do so, may lead to the termination of this Agreement.

## Term of Agreement:

This Purchase Order is effective beginning 3/4/2016 and expiring on 12/31/2018.

### Note:

The Delivery Date shown is the end date of this Purchase Order. The Validity End Date is an internal date that is used for Dominion accounting purposes only.

### Supplemental Invoice and Payment Instructions:

In addition to the provision for "Invoices and Payment" in the attached Terms and Conditions, Supplier shall comply with the following instructions when submitting invoices for payment:

All invoices shall reference this Purchase Order number.

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 4 of 13 RICHMOND VA 23227

All invoices shall be submitted in accordance with the terms of the applicable "Invoices and Payment" provision in the attached Terms and Conditions, or if not specified therein, within thirty (30) days of the completion date of Supplier's performance.

Invoices should be submitted electronically through Purchaser's Invoicing and Payment Portal (provided by Taulia at portal.taulia.com). If you have not enrolled in Taulia, please request an invitation by visiting

https://www.dom.com/corporate/doing-business-with-us/suppliers/active-suppliers

Then, click on the link "Please click here to request an invitation."

When submitting invoices electronically, do not duplicate with paper invoices.

Invoices submitted on paper should be mailed to the address shown on the front page and listed below:

Attn: Accounts Payable Dominion Virginia Power PO Box 25459

Richmond, VA 23260-5459

Payment will be made in accordance with the specified "Invoices and Payment" provision in the attached Terms and Conditions, provided that:

- The invoice is received at the correct address
- The invoice is complete and accurate
- All documentation to support compensable expenses (e.g. time sheets, expense reports, travel receipts, price sheets, sub-supplier invoices, etc.) are submitted with the invoice. If submitting invoices electronically, attach documentation in PDF format.

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 5 of 13 RICHMOND VA 23227

- The invoice is deemed acceptable by Purchaser's authorized representative

Unless otherwise directed or requested by Purchaser's Supply Chain representative, Supplier shall ensure that all invoicing for all work scope is submitted on a prompt and timely basis. Delayed invoice submission adversely affects Purchaser's budgeting and administrative controls and may result in delayed payment to Supplier.

For invoice payment inquiries, contact Accounts Payable directly at (804)771-6200 or email your request to the following e-mail address: ap invoices hotline@dom.com.

Suppliers are strongly encouraged to enroll in Purchaser's Invoicing & Payment portal to check their invoice's payment status at http://portal.taulia.com. To get more information or enroll, please visit:

https://www.dom.com/suppliers/active-suppliers/invoicing-and-payment-portal.jsp

Pricing Adjustment Provisions:

Purchaser shall not be obligated to pay any rates other than those shown herein.

Foreign Material Exclusion (FME):

Supplier will comply with the following FME practices along with any additional FME requirements included in the Scope of Work herein and/or subsequent Purchase Order releases.

Purchaser is committed to operating power stations free from introduction of any material that is not part of the system or component as designed. Purchaser's expectation is that the Supplier verifies, prior to performing any service, shipment of material, or use of any equipment, that the work will not introduce dirt, debris, broken or missing parts, oil, slag, tools, rags, chemicals, machine tailings, lapping compounds, polishing wires, tie wires, solvents, grinding particles, paint chips, leak-sealing compounds, personal

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 6 of 13 RICHMOND VA 23227

protective equipment, and any other items that could adversely affect the intended operation, components, or chemistry of the system. Purchaser additionally requires that the Supplier identify any items installed internally for shipping purposes that are to be removed for operation of the equipment.

Material Shipping Instructions:

\*F O B - Destination, Frt. Collect or Prepaid and Add

## Jobsite Requirements:

Supplier's personnel shall report to and direct all jobsite/ work related questions to the Purchaser's jobsite representative designated at the time services are requested. The site may require a pre-job briefing, safety orientation, or other site specific requirements. Contact the Authorized Station Contact(s) listed herein for all site specific requirements.

If requested by the site for time and material work, Supplier shall be responsible for filling out Purchaser's time sheets to document all time worked on site. Prior to leaving the site each day, the Supplier shall be responsible for submitting accurate time sheets to Purchaser's jobsite representative for review. Purchaser shall review and approve or reject Supplier time sheets in a timely manner. Supplier shall retain a copy of the signed time sheet and submit as supporting documentation with any invoice(s).

### Correspondence:

All technical correspondence pertaining to this Agreement shall be sent to the Authorized Station Contact(s) listed herein.

All correspondence on commercial or contractual considerations pertaining to this Agreement shall be sent to:

Kevin Brannan

kevin.e.brannan@dom.com

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 7 of 13 RICHMOND VA 23227

If a signed copy of this Purchase Order is desired by Supplier, please indicate on the acceptance copy. Otherwise, both parties agree that neither will raise any defense of lack of writing, lack of signature or any other similar defense based upon a "Statute of Frauds" or similar rule in any dispute that may arise between them over an electronic transmittal.

ltem Material # Order qty.	Unit	Description Price per unit	Net value
00001	Each	Bremo CCR Impoundmen	Closures

### TAX IS NOT APPLICABLE

Rel. ord. against contract 46027533 Item 00001

### Scope of Work:

Supplier shall provide the necessary labor, materials, consumables, equipment, engineering, analysis and reporting required to perform the required CCR Closure sampling, set forth in the Individual VPDES Permit for Bremo Power Station No. VA0004138. Laboratory space will be provided by Bremo Power Station, to include access to fume hood, argon gas, and other electrial power supplies.

Supplier shall provide sampling services whereas the samples will be taken and returned in proper timeframe to support analysis and reporting by the Station to the VADEQ under the requirements set forth in the new Individual VPDES Permit. Additional Process Samples taken and anlyzed at the request of the Site or Dominion Power Generation Environmental.

Supplier will utilize PACE Laboratories as their subcontractor for taking/analyzing the samples. If it becomes necessary to replace this subcontractor, Dominion requires a minimum of thirty days notice. The on-site lab work will be 24 hours a day, 7 days a week. One chemist per

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 8 of 13 RICHMOND VA 23227

Item	Material #	Description		
10111	Widtorial II	Description		
Order (	atv.	Unit	Price per unit	Net value
	17.	••	i noo por anii	Not value

12 hour shift shall be supplied. Supplier shall ensure Laboratory Oversite is Supplied and that requirements are met. Supplier Project Manager shall be specified to Dominion and shall ensure that station reporting requirements are met.

## Specific sample requirements:

Weekly 500-Series VPDES sampling, analysis, and reporting for Outfall 504 including:

- Three sampling events each week, including labor and equipment, with one 4-hour composite sample collected during each event, with at least 48 hours between sampling events. In addition, an Equipment Blank will be prepared and analyzed once each week to evaluate possible field contamination. If a sample cannot be obtained due to the absence of flow, "no discharge" documentation will be provided.
- On-site laboratory analysis through Pace Analytical Services, Inc. (Pace) with next-day by 12-noon turnaround time (TAT) for the weekly compliance analyses.
- Continuous 24-hour on-site laboratory analysis through Pace for process sampling for metals analysis. Pace's on-site laboratory will conduct metals analysis for both process samples and compliance samples.
- Dedicated courier service through Pace with daily 12-noon pick-up time for those analyses required for compliance which cannot be run in the on-site laboratory.
- Data quality control review and reporting to Dominion within 24 hours of receipt of laboratory results.
- -Data entry and maintenance in Supplier's EQuIS data management system.

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Item	Material #	Des	cription						
Order	qty.	Unit	Price per unit	Net value					

- Electronic delivery of tabulated laboratory results with permit limit comparisons, and;
- Program management and environmental compliance consulting assistance.

## Authorized Station Contact(s):

The following individual(s) are the primary contacts for work done at the site and are authorized to release goods and/or services under the provisions of this Purchase Order.

Contact Information:

Site Contact = Maria Gwynn 804-921-4107

Technical Contact =
Ken Roller 804-273-3494
Mike Surface 804-273-2530

Authorized to Release Work (#1) Maria Gwynn 804-921-4107 Authorized to Release Work (#2) Michael Glagola 804-273-2362

The designation as "Authorized Station Contact" shall not be construed by Supplier to confer upon the contact(s) an actual or apparent authority to act as Agent for Purchaser except to issue said verbal or written requests or work direction consistent with the terms of this Agreement. These personnel shall have no authority to amend, alter or otherwise modify the scope of work or any of the terms and conditions

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Item Material # Description
Order qty. Unit Price per unit Net value

of this Purchase Order.

### Compensation

All work shall be performed in accordance with the following rates:

- Weekly 500-Series VPDES sampling, analysis, and reporting for Outfall 504

Estimated hours = 225

Estimated labor = \$25,950.00

Estimated expenses = \$111,200.00

Monthly Not to Exceed = \$137,150.00

- Monthly 500-Series VPDES sampling, analysis, and reporting for Outfall 504

Estimated hours = 52

Estimated labor = \$5,550.00

Estimated expenses = \$3,700.00

Monthly Not to Exceed = \$9,250.00

As-needed Sampling Assistance for Non-500-Series Outfalls site VPDES

Estimated hours = 43

Estimated labor = \$5,400.00

Estimated Expenses = \$200.00

Monthly Not to Exceed = \$5,600.00

## Total Monthly Budget

Estimated hours = 290

GOLDER ASSOCIATES INC PO number/date
2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016
RICHMOND VA 23227

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Item Material # Description
Order qty. Unit

Price per unit

Net value

Estimated labor = \$36,900.00

Estimated expenses = \$115,100.00

Total Monthly Budget = Not to exceed \$152,000.00

## Alternate Pricing Structure:

For billing purposes only, and if requested by Purchaser, Supplier shall provide fixed price quotations for services/materials as released by the Authorized Station Contacts named herein. Supplier is authorized to bill based on the station contact's approval of the corresponding applicable quotation. The scope of work for Work performed on this order shall be mutually agreed upon by the parties.

## Performance Schedule:

This order is valid for services through 12/31/2018 unless otherwise modified by written change order.

GOLDER ASSOCIATES INC PO number/date Page 2108 2108 W LABURNUM AVE SUITE 200 70300442 / 03/04/2016 12 of 13 RICHMOND VA 23227

The item covers the following services:
1 8000038 Value Limit PO

1 EA

Released from the line 1

This company has valid Direct Pay permits issued by the North Carolina and West Virginia Taxing Authorities. Under the authority of these permits, this company self-assesses applicable state and local sales and use tax for all purchases delivered to the states of North Carolina and West Virginia. Included in this purchase order is a copy of any applicable Direct Pay permit to be provided to supplier. Supplier should not charge sales taxes on purchases delivered to West Virginia or North Carolina.

NOTE TO SUPPLIERS DELIVERING PURCHASES TO DOMINION VIRGINIA POWER IN THE COMMONWEALTH OF VIRGINIA: Effective October 1, 2005 Dominion Virginia Power's temporary Direct Pay authority issued by the Virginia Department of Taxation expires. Supplier should charge Virginia state and local sales tax, if applicable, on all invoices issued after October 1, 2005 for purchases delivered to Virginia.

NOTE TO CONTRACTORS PERFORMING REAL PROPERTY CONSTRUCTION SERVICES TO VIRGINIA POWER AT LOCATIONS IN VIRGINIA: If supplier is a contractor providing materials and/or services in conjunction with a real property construction project located in the Commonwealth of Virginia, supplier shall invoice Dominion Virginia Power separately, as a separate line item on their invoice, for the reimbursement of any sales and/or use taxes incurred by supplier on the purchase of tangible personal property formerly exempt from Virginia sales tax under Virginia Code Section 58.1-610(B) and facilitated by Virginia form ST-11A. Such separate line item shall include the description, "Sales Tax Paid by Contractor on Purchases Formerly Exempted by Virginia Form St-11A (Public Service Company Exemption).

## Direct Pay Permit numbers:

Dominion North Carolina Power - NC permit number - 00148 Dominion West Virginia Power - WV permit number L1368364032

Buyer's PURCHASE ORDER NO. and ITEM NO. MUST appear on ALL invoices, shipping papers, packages and correspondence.

GOLDER ASSOCIATES INC PO number/di 2108 2108 W LABURNUM AVE SUITE 200 7030044		Page 13 of 13
RICHMOND VA 23227	.2 / 00/04/2010 .	15 01 15
Within 10 days of the date of issuance of this order or revision	<i>A</i> /1	ts acceptance
by signing and returning the original. Refer to Contact Person'		13
Accepted By: Date: 3-8-10	By: Alley	3-8-16
Supplier S Sanussan VI	Furchase	r
Supplier R. D. FRANCESCO, W.	$\mathcal{O}$	

GOLDER ASSOCIATES INC

At Golder Associates we strive to be the most respected global group of companies specializing in ground engineering and environmental services. Employee owned since our formation in 1960, we have created a unique culture with pride in ownership, resulting in long-term organizational stability. Golder professionals take the time to build an understanding of client needs and of the specific environments in which they operate. We continue to expand our technical capabilities and have experienced steady growth with employees now operating from offices located throughout Africa, Asia, Australasia, Europe, North America and South America.

Africa + 27 11 254 4800
Asia + 852 2562 3658
Australasia + 61 3 8862 3500
Europe + 356 21 42 30 20
North America + 1 800 275 3281
South America + 55 21 3095 9500

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Fax: (724) 935-6416

